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**A CHEMICAL CHARACTERIZATION AND
PERFORMANCE STUDY OF PHOTODECOMPOSED
HEXANITROSTILBENE (HNS) AND
HEXANITROBIBENZYL (HNBiB)**

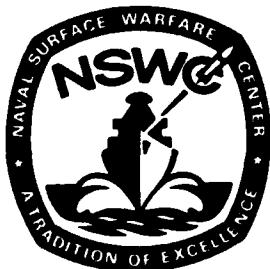
BY ELEONORE G. KAYSER

RESEARCH AND TECHNOLOGY DEPARTMENT

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REPORT DOCUMENTATION PAGE

| | | | | | |
|---|----------|---|---|---|----------------------------------|
| 1a. REPORT SECURITY CLASSIFICATION UNCLASSIFIED | | | 1b. RESTRICTIVE MARKINGS | | |
| 2a. SECURITY CLASSIFICATION AUTHORITY | | | 3. DISTRIBUTION/AVAILABILITY OF REPORT | | |
| 2b. DECLASSIFICATION DOWNGRADING SCHEDULE | | | Approved for public release; distribution is unlimited. | | |
| 4. PERFORMING ORGANIZATION REPORT NUMBER(S) NSWC TR 90-60 | | | 5. MONITORING ORGANIZATION REPORT NUMBER(S) | | |
| 6a. NAME OF PERFORMING ORGANIZATION Naval Surface Warfare Center | | 6b. OFFICE SYMBOL (If applicable) R16 | | 7a. NAME OF MONITORING ORGANIZATION | |
| 6c. ADDRESS (City, State, and ZIP Code) White Oak Laboratory 10901 New Hampshire Avenue Silver Spring, MD 20903-5000 | | | | 7b. ADDRESS (City, State, and ZIP Code) | |
| 8a. NAME OF FUNDING/SPONSORING ORGANIZATION NASA/LBJ Space Center | | 8b. OFFICE SYMBOL (If applicable) | | 9. PROCUREMENT INSTRUMENT IDENTIFICATION NUMBER | |
| 8c. ADDRESS (City, State, and ZIP Code) 2101 Webster-Seabrook Road Houston, TX 77058 | | | | 10. SOURCE OF FUNDING NOS. | |
| | | | | PROGRAM ELEMENT NO. T-8268J | PROJECT NO. |
| | | | | TASK NO. | WORK UNIT NO. |
| 11. TITLE (Include Security Classification) A Chemical Characterization and Performance Study of Photodecomposed Hexanitrostilbene (HNS) and Hexanitrobibenzyl (HNBiB) | | | | | |
| 12. PERSONAL AUTHOR(S) Kayser, Eleonore G. | | | | | |
| 13a. TYPE OF REPORT Final | | 13b. TIME COVERED FROM 1/84 TO 12/84 | | 14. DATE OF REPORT (Yr., Mo., Day) 1989, August | |
| 15. PAGE COUNT 62 | | | | | |
| 16. SUPPLEMENTARY NOTATION | | | | | |
| 17. COSATI CODES | | | 18. SUBJECT TERMS (Continue on reverse if necessary and identify by block number) | | |
| FIELD | GROUP | SUB. GR. | | | |
| 7 | 5 | | | | |
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| | | | | | |
| 19. ABSTRACT (Continue on reverse if necessary and identify by block number) | | | | | |
| <p>The photolysis of 2,2'4,4'6,6'-hexanitrostilbene (HNS) and 2,2'4,4'6,6'-hexanitrobibenzyl (HNBiB) has been investigated. This laboratory study was carried out at wavelengths (λ) greater than 280 nanometers (nm) using sunlight and a #7740 pyrex filtered, 400 watt, Hanovia medium pressure, mercury (Hg) arc source.</p> <p>The methods and conditions under which HNS and HNBiB are decomposed photolytically are presented as well as the isolation and identification of several of the more stable photoproducts. The characterization methods include: (1) thin-layer chromatography (TLC), (2) color macrophotography, (3) scanning electron microscopy (SEM), (4) nuclear magnetic resonance (NMR) spectroscopy, (5) high-performance, liquid chromatography (HPLC), and (6) vacuum thermal stability (VTS). Velocity</p> | | | | | |
| 20. DISTRIBUTION/AVAILABILITY OF ABSTRACT | | | 21. ABSTRACT SECURITY CLASSIFICATION | | |
| <input checked="" type="checkbox"/> UNCLASSIFIED/UNLIMITED <input type="checkbox"/> SAME AS RPT <input type="checkbox"/> DTIC USERS | | | UNCLASSIFIED | | |
| 22a. NAME OF RESPONSIBLE INDIVIDUAL Eleonore G. Kayser | | | 22b. TELEPHONE NUMBER (Include Area Code) (202) 394-2860 | | 22c. OFFICE SYMBOL R16 |

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and output measurements were also obtained on the irradiated HNS material which was loaded into small diameter, explosive train hardware [e.g., shielded mild detonating cord (SMDC) and flexible linear shaped charge (FLSC)]. The SMDC explosive component consists of a silver-sheathed cord with an HNS core load of 2-1/2 grains/foot. The FLSC component is also silver sheathed and contains an HNS core load of 20 grains/foot.

The irradiated samples of HNS and HNBiB contained numerous organic compounds. Five photodecomposition products were separated and identified from the brown material extracted from the surface of the photolyzed HNS. These included: 1,3,5-trinitrobenzene; 4,6-dinitro[2,1]benzoxazole; 2,4,6-trinitrobenzaldehyde; 4,6-dinitroisoanthranil; and 2,4,6-trinitrobenzoic acid. The five products amounted to approximately 10 percent by weight of the photodecomposition products. The remaining 90 percent defied separation and identification. Seven photodecomposition products were identified from the irradiated HNBiB samples. These include: 2,4,6-trinitrobenzaldehyde; 1,3,5-trinitrobenzene; 2,4,6-trinitrotoluene; 4,6-dinitro[2,1] benzisoxazole; 4,6-dinitroisoanthranil; 2,4,6-trinitrobenzyl alcohol; and HNS. Several of the single-ring, benzene soluble photoproducts, i.e., 2,4,6-trinitrobenzaldehyde, 2,4,6-trinitrobenzyl alcohol, and the 2,4,6-trinitrobenzoic acid, were found to be more photolabile than either the HNS or the HNBiB.

The photolytic conditions (both sunlight and Hg lamp) used in this work are beyond those which HNS is subject to under normal synthesis and handling operations. The results of this study indicate that the SMDC's (2-1/2 grains/foot) fabricated with the irradiated HNS material performed satisfactorily even after additional thermal exposure to 350°F/50 hours. However, 100 percent of the SMDC lines containing the irradiated HNS failed after thermal exposure to 375°F/50 hours. None of these failures occurred in the booster tips but they occurred randomly along the SMDC lines. The results also indicate that additional thermal exposure to 375°F/50 hours appeared to have no significant effect on performance of the FLSC lines (20 grains/foot) containing irradiated HNS.

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EXECUTIVE SUMMARY

The objective of this research was two-fold. First, a laboratory investigation of the photodecomposition of 2,2',4,4',6,6'-hexanitrostilbene (HNS) and 2,2',4,4',6,6'-hexanitrobibenzyl (HNBiB) at wavelengths (λ) greater than 280 nanometers (nm) was performed. For this study, two light sources were used--natural sunlight and a #7740, pyrex filtered, 400 watt, Hanovia medium pressure, mercury (Hg) arc source which simulates natural sunlight only at a greater intensity. Second, an investigation of the effects of this irradiated HNS material, fabricated into SMDC and FLSC lines, on detonation cord performance was conducted. The methods and conditions used to photolytically decompose the HNS and HNBiB, as well as the isolation and identification of several of the relatively stable photoproducts, are discussed. The characterization techniques include: (1) thin-layer chromatography (TLC), (2) color macrophotography, (3) scanning electron microscopy (SEM), (4) nuclear magnetic resonance (NMR) spectroscopy, (5) high-performance liquid chromatography (HPLC), and (6) vacuum thermal stability (VTS). Detonation velocity measurements were obtained on the explosive train hardware (e.g., shielded mild detonating cords (SMDC's) and flexible linear shaped charges (FLSC). These lines were fabricated at Explosive Technology (ET), Fairfield, California, using the HNS irradiated at the Naval Surface Warfare Center, White Oak Laboratory, Silver Spring, Maryland. Output measurements were also obtained on the small diameter SMDC lines. The SMDC is a silver-sheathed cord containing an HNS-II cord load of 2-1/2 grains/foot. This line is capped at both ends with a stainless steel booster tip containing 65 mg of HNS-I. The FLSC component is also silver sheathed and contains an HNS-II core load of 20 grains/foot.

The irradiated samples of HNS and HNBiB contain numerous organic compounds. Five photodecomposition products were separated and identified from the brown material extracted from the surface of the photolyzed HNS. These included: 1,3,5-trinitrobenzene; 4,6-dinitro[2,1,1]benzisoxazole; 2,4,6-trinitrobenzaldehyde; 4,6-dinitroisoanthranil; and 2,4,6-trinitrobenzoic acid. The five compounds amounted to approximately 10 percent by weight of the HNS photodecomposition products. The remaining 90 percent contained at least six, and possibly ten, other compounds (as evidenced by HPLC peaks and TLC spots); however, identification was not possible due to (1) the trace quantities that were formed and (2) the instability of the materials. From the remaining dark brown tacky fraction, a waxy oil and/or waxy solid was also extracted but could not be identified. This extracted sample contained high molecular weight and/or polar compounds, since most of the sample (~70 percent) remained at the origin when chromatographed with 100 percent methanol.

Seven photodecomposition products were identified from the irradiated HNBiB samples. These include: 2,4,6-trinitrobenzaldehyde; 1,3,5-trinitrobenzene; 2,4,6-trinitrobenzyl alcohol; and HNS. Several of the single-ring benzene soluble photoproducts, i.e., 2,4,6-trinitrobenzaldehyde, 2,4,6-trinitrobenzyl alcohol, and the 2,4,6-trinitrobenzoic acid were found to be more photolabile than either the HNS or the HNBiB.

The photolytic conditions (both sunlight and Hg lamp) used in this study are beyond those which HNS is subject to under normal synthesis and handling operations. The results of this study indicate that the SMDC's (2-1/2 grains/foot) fabricated with the irradiated HNS material performed satisfactorily after additional thermal exposure to 350°F/50 hours. However, 100 percent of the SMDC lines containing the irradiated HNS failed after thermal exposure to 375°F/50 hours. None of these failures occurred in the booster tips but occurred randomly along the SMDC lines. The results also indicate that additional thermal exposure to 375°F/50 hours appeared to have no significant effect on the performance of the FLSC lines (20 grains/foot) containing irradiated HNS.

FOREWORD

This report contains chemical, photographic, and performance data generated on 2,2',4,4',6,6'-hexanitrostilbene (HNS) and 2,2',4,4',6,6'-hexanitrobibenzyl (HNBiB) photolyzed at wavelengths (λ) greater than 280 nanometers (nm). This study was funded by the National Aeronautics and Space Administration, Lyndon Baines Johnson (NASA-LBJ) Space Center, Houston, Texas, under NASA Defense Purchase request No. T-8268J over the period 1 January 1984 to 31 December 1984 to ensure that HNS used in any man-rated system will adequately execute its life-critical function.

This report describes the methods and conditions under which HNS and HNBiB were decomposed photolytically and the isolation and identification of several of the resultant photoproducts.

The identification of commercial materials and/or manufacturers implies neither criticism nor endorsement by the Naval Surface Warfare Center.

The author wishes to thank the very patient loading operators at Explosives Technology, Fairfield, California, Dr. Marriner K. Norr for his contribution in the form of the scanning electron micrographs, and Mr. Bryan A. Baudler for the performance data.

Approved by:



WILLIAM H. BOHLI, Head
Energetic Materials Division

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CHAPTER 1

INTRODUCTION

The Naval Surface Warfare Center, White Oak Laboratory (NSWC/WO), Silver Spring, Maryland, was contracted by the National Aeronautics and Space Administration, Lyndon Baines Johnson (NASA-LBJ) Space Center, Houston, Texas, to conduct a laboratory study of the photochemistry of HNS and HNBiB, the major impurity in the HNS synthesis process. The objectives were to investigate (1) the photodecomposition of 2,4',4',6',6'-hexanitrostilbene (HNS) and 2,2',4',4',6',6'-hexanitrobibenzyl (HNBiB) at wavelengths (λ) greater than 280 nanometers (nm) and (2) the effects of this irradiated HNS material, fabricated into SMDC and FLSC lines, on detonation cord performance. The photodecomposition was carried out using two light sources, natural sunlight and a #7740 pyrex filtered, 400 watt, Hanovia medium pressure mercury (Hg) arc source which simulates natural sunlight only at a greater intensity.

When compared to currently used explosives such as RDX, HMX and TNT, HNS is considered to be insensitive to electrostatic discharge, impact, and heat and has proven to be an exceptionally good explosive for high temperature applications in shielded mild detonating cords (SMDC) and flexible linear shaped charges (FLSC). These lines are resistant to initiation by lightning or weapon projectiles and will not build to detonation in a fire but will melt and burn. HNS has found numerous applications throughout the aerospace industry in explosive-actuated devices used in man-rated systems such as the personnel escape systems of high-speed aircraft (e.g., F-4, F-111, F-16, B-1B, F-14, F-18) and spacecraft. Rigid explosive transfer lines containing HNS are also used in missiles such as the Harpoon, Trident, ALCM, Peacemaker, and ACM.

HNS and HNBiB, as solids and in solution, become colored when exposed to sunlight and/or room light. Both HNS and HNBiB undergo a series of color--changes (light yellow-->orange brown) over a period of several hours exposure to sunlight. For this reason, the following study was undertaken to determine what effect photolysis has on the performance of transfer lines containing photolyzed HNS. The technical approach involved five phases:

Phase I--Establish standards of comparison for all subsequent samples.

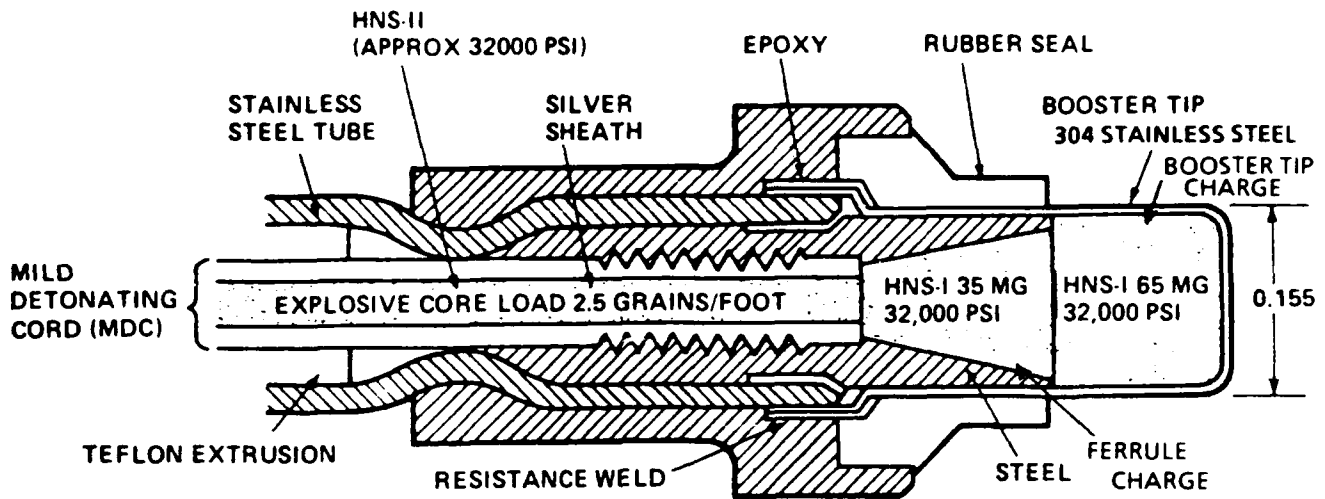
Phase II--Collect kinetic data on the rates of photolysis of HNS and HNBiB in fused quartz glassware irradiated using sunlight and a 450 watt, Hanovia medium pressure, Hg arc source.

Phase III--Isolate and identify the major photoproducts using appropriate analytical techniques.

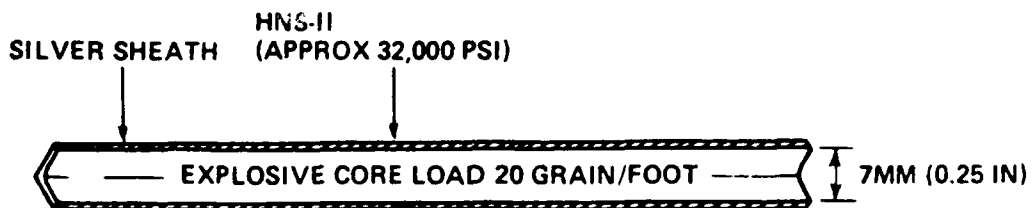
Phase IV--Fabricate silver-sheathed detonating cords, SMDC's and FLSC's (Figure 1A and 1B) containing HNS-I and HNS-II material as received and after photolysis with sunlight and the 450 watt, Hanovia medium pressure, Hg arc source.

Phase V--Conduct thermal degradation studies using the fabricated lines to determine the limit at which line functionality is maintained.

Phase I, II, III, and V were carried out at NSWC-WO, while the cords (Phase IV) were fabricated at Explosive Technology (ET), Fairfield, California.



(A) CROSS SECTION OF RIGID EXPLOSIVE TRANSFER LINE (1 GRAIN--65 MG)



(B) CROSS SECTION OF FLEXIBLE LINEAR SHAPED CHARGE

FIGURE 1. CROSS SECTIONS OF RIGID EXPLOSIVE TRANSFER LINE AND FLEXIBLE LINEAR SHAPED CHARGE

CHAPTER 2

EXPERIMENTAL

EXPLOSIVE MATERIALS

Standards of HNS-I, HNS-II, and HNBiB were prepared by the synthesis methods reported in Reference 1. These standards were recrystallized until a purity of 99.8 percent was obtained for HNS-I and HNS-II and a purity of 99.7 percent was obtained for the HNBiB. The values were confirmed by thin-layer chromatography (TLC) (2-dimensional) using an ethylene diamine spray reagent and high-performance liquid chromatography (HPLC). These reference samples were then used in Phase II and III of this study. The bulk HNS-I and HNS-II, used in Phase IV and V, was furnished by the NASA/LBJ Space Center, Houston, Texas. The HPLC assay data and manufacturers identification lot numbers of these materials can be found in Table 1.

TABLE 1. IDENTIFICATION AND HPLC ASSAY DATA OF HNS-I AND HNS-II, AS RECEIVED

| NSWC Ident # | HNS Type | HNS Mfg/Lot # | Year Produced or Converted* | HPLC Assay | |
|-----------------|----------|-----------------------|--------------------------------|------------|---------|
| | | | | % HNS | % HNBiB |
| 1987 | I | Chemtronics/ 105-6 | 1975 P | 99.3 | 0.6 |
| 1556 | II | Chemtronics/ 36-45 | 1971 C | 99.8 | 0.2 |

*P = Year HNS-I produced by Chemtronics

C = Year HNS-I converted to HNS-II at Chemtronics

PHOTOLYSIS APPARATUS

Irradiation of solid HNS and HNBiB was carried out with a 450 watt, Hanovia medium pressure, mercury arc lamp (Hg lamp) housed in a #7740 pyrex cooling jacket. To a first approximation, this apparatus duplicates solar radiation but with greater intensity. The #7740 pyrex cooling jacket permits light at wavelengths longer than 280 nm to be transmitted to the samples. By comparison, the earth's atmosphere absorbs virtually 99 percent of the radiation below 280 nm emitted by the sun.²

MATERIAL PHOTOLYSIS

Milligram quantities of reference HNS and HNBiB were dusted on a glass slide positioned 4 cm from the Hg lamp. These samples were irradiated for 5-, 10-, 15-, and 30-minute periods. Ninety such runs were consolidated to obtain information for Phase III of this study. Concentrated solutions of the reference materials in acetonitrile and dimethyl sulfoxide (DMSO) (total volume about 10 mL) were photolyzed in fused quartz glassware positioned 4 cm from the Hg source, housed in a pyrex cooling jacket. These solutions were stirred constantly during the irradiation period to obtain consistent kinetic data. Duplicate samples of HNS and HNBiB, as solids and in solution, were also irradiated in sunlight. Thirty such runs were consolidated for Phase III of this study.

The HNS-I and HNS-II needed for Phase IV of this study were irradiated in a quartz tube (5 cm outside diameter by 21 cm long) which was continuously rotated at a slow speed in a horizontal position (1) in direct sunlight (no visible cloud cover) for 4 hours and (2) 3.5 cm from the 450 watt, Hanovia Hg lamp, housed in a #7740 pyrex cooling jacket, for a 30-minute period.

THIN-LAYER CHROMATOGRAPHY (TLC)

The thin-layer chromatographic support was Merck Silica Gel 254 HF 60-G. Silica Gel 60-G was used for column chromatography (column grade). Thin-layer chromatograms³ of some of the HNS and HNBiB photodecomposition products were visualized by spraying the plates with ethylene diamine/dimethyl sulfoxide (2:1) (v,v) (EDA/DMSO) reagent or neat EDA. Polynitro-aromatic compounds form intense, brightly colored Meisenheimer-type complexes with this reagent.⁴ Identification of several components present in the solvent fractions was obtained by comparison of the R_f values of the known compounds with the R_f of the unknown developed concurrently on the same plate.

NUCLEAR MAGNETIC RESONANCE (NMR) SPECTROSCOPY

The NMR spectra were obtained on a Varian XL-200 fourier transform NMR spectrometer. All chemical shifts are reported in hertz downfield from the standard reference material tetramethylsilane (TMS). The letters and numbers in parentheses refer to line multiplicity and integrated areas, respectively. Some of the photolysis products were identified by comparison of the NMR spectra with those of authentic samples.

HIGH-PERFORMANCE LIQUID CHROMATOGRAPHY (HPLC)

A high-performance liquid chromatograph (Waters Associates Model ALC 202) equipped with a 254 nm wavelength detector, a solvent delivery system (Model 6000), and a U6K high-pressure loop injector was used with a Model RCM-100 module containing a reverse phase C-18 Radial-Pak cartridge. Sample solutions were eluted isocratically at ambient temperature. Column flow was 0.5 mL/minute, with the mobile phase consisting of HPLC grade methanol and distilled water, 70:30 (v,v). The solvent mixtures were filtered, but not degassed prior to HPLC analysis, and sample injections of 2-10 μ L were used.

Several of the HNS and HNBiB photolysis products were identified, when possible, by comparing the LC retention times with those of authentic samples.

COLOR MACROPHOTOGRAPHY

Color photographs (15-25x magnification) were taken of cross sections of the SMDC booster tips. The booster tip (Figure 2) was dissected with a tubing cutter at the ferrule charge-to-booster charge plane. The cup was cut and broken open to minimize any physical disturbance of the pressed explosive. Color photos of the cord were taken of a 2-inch section which was opened longitudinally with a file. The SMDC cord (Figure 2) was cut with a tubing cutter approximately 4 cm from each end of the line. Photos were obtained with a Wild microscope (M-400) made in Heerbrugg, Switzerland; KodaColor VR ASA 400, 35 mm film was used for documentation.

To obtain macrophotographs of the HNS contained in the FLSC, the metal sheath was filed down to the explosive fill (Figure 2), and the sheath was then peeled back to expose the explosive core. Using this method, the entire surface of the explosive core of the chevron-shaped charge could be visually inspected without damaging the integrity of the explosive.

SCANNING ELECTRON MICROSCOPY (SEM)

SEM photos were obtained with an ARRAY, Model 1000A, scanning electron microscope (5000x magnification). The SEM data were processed on a cathode ray tube (7-inch diagonal, 2500-line resolution) and photographed with a Polaroid camera. Sample preparation included vacuum sputter-coating with gold.

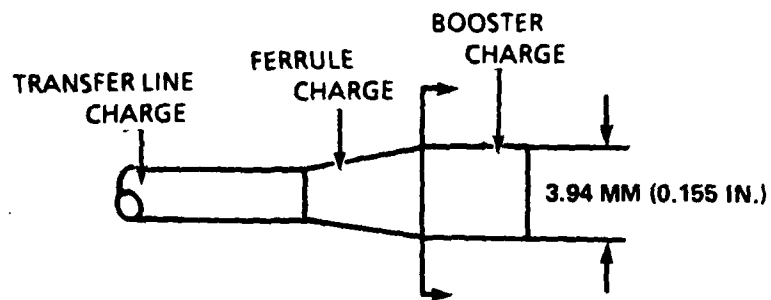
PERFORMANCE

Performance⁵ was determined by (1) detonation velocity measurements and (2) the output dent from an explosive pellet initiated by the SMDC tip.

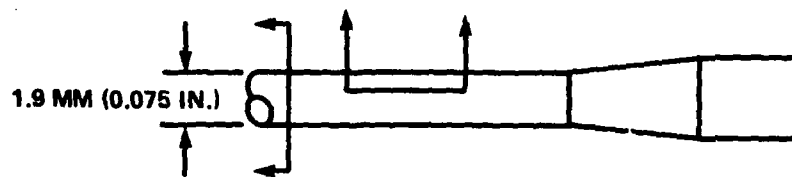
Both the SMDC and FLSC lines were initiated by E1A (#6) blasting caps. Detonations with velocity measurements (Figure 3) of the SMDC charges were obtained using two ionization probes, one taped at the detonator/cord interface and the other at the output end of the cord. A Hewlett-Packard timer was used to measure the transfer times between these probes. Steel blocks (1.25-inch width x 1.25-inch length x 0.625-inch depth) were used to measure output results from the booster detonation transfer. The performance of the end booster is determined by its ability to initiate an explosive (CH-6) pellet.⁶

Detonation velocity measurements (Figure 4) of the FLSC charges were calculated at two different points on each line. Four ionization probes were placed at regular intervals on each line where a slight notch had been cut to expose the HNS-II. These probes were connected to two separate timers. Velocities were calculated based on the distance between probes. Each line was placed on an aluminum plate when fired. Complete initiation of the line was determined by subsequent inspection of the slot cut into the plate.

SMDC



(A) BOOSTER TIP



(B) TRANSFER LINE

FLSC

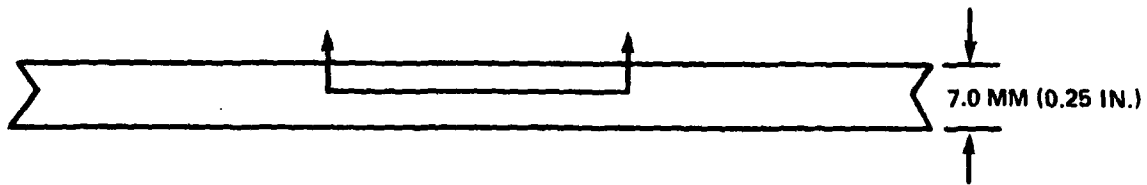


FIGURE 2. SMDC AND FLSC DISSECTION SITES FOR PHOTOGRAPHIC AND CHEMICAL ANALYSIS

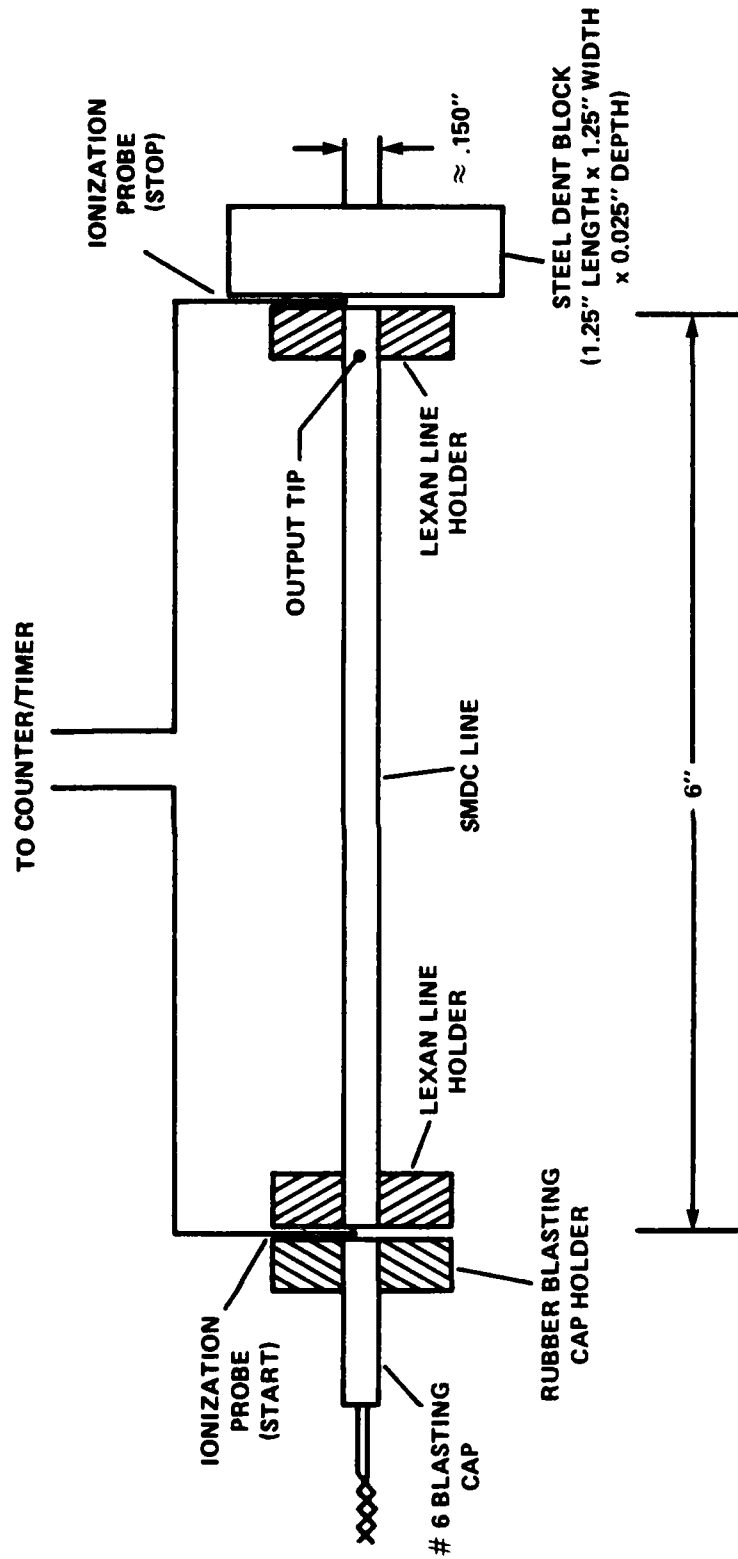


FIGURE 3. DETONATION VELOCITY TEST SETUP (SMDC LINES)

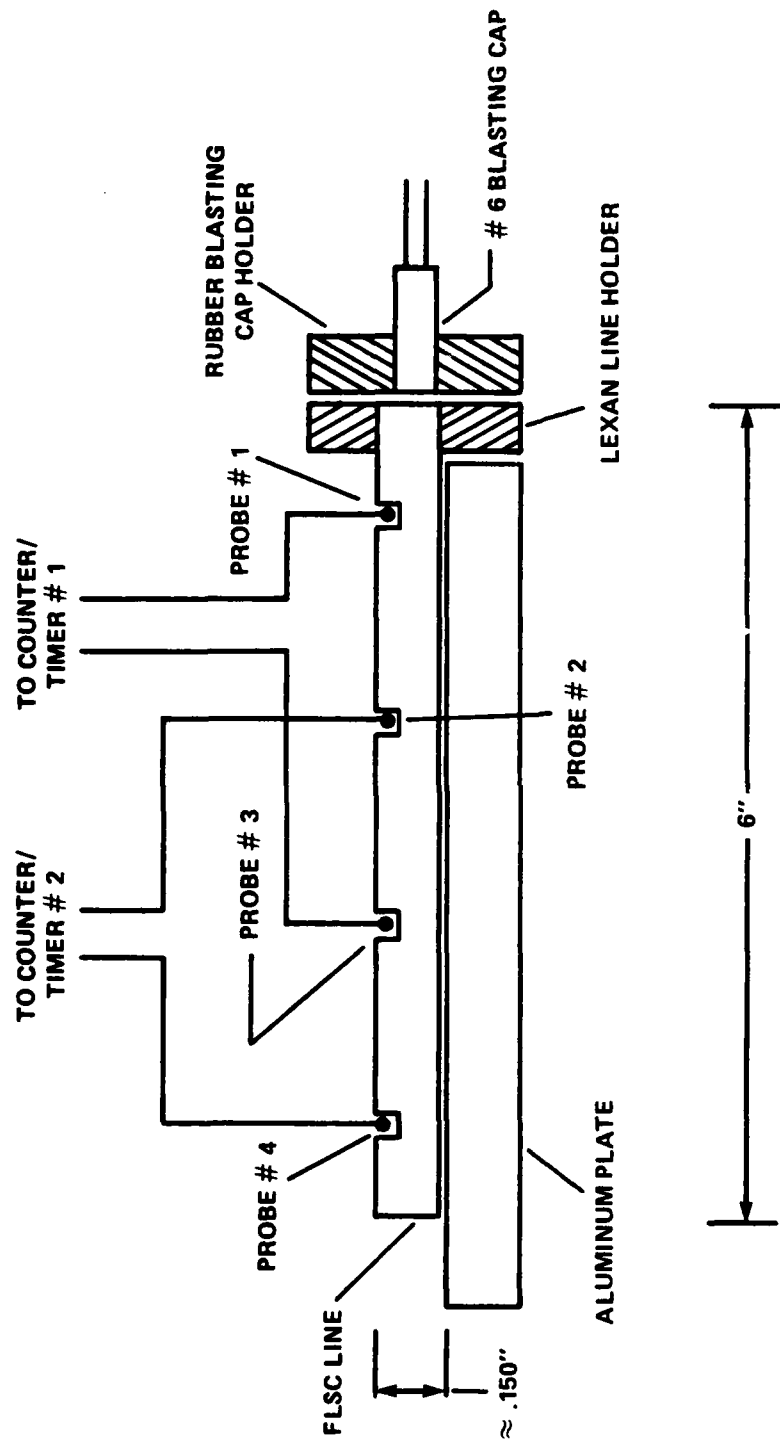


FIGURE 4. DETONATION VELOCITY TEST SETUP (FLSC LINES)

HPLC ANALYSIS

BOOSTER TIP--SMDC

For the HPLC analyses, HNS-I was removed from the cup and the material was mechanically blended. The samples were weighed on a Mettler microbalance (standard deviation $\pm 1.0 \mu\text{g}$) and then dissolved in DMSO solvent. Two to three HPLC analyses were carried out on each BT. The analyses were reproducible to within ± 3 percent.

TRANSFER LINE--SMDC

Several 1 to 2 mm cross sections were cut from the cord for the HPLC analyses. DMSO was added to the silver (Ag) sheathed line sample. The solution was placed in an ultrasonic bath at room temperature for 30 minutes to completely dissolve the HNS-II material. The quantity of material was determined by the tare in weighing the original and the empty cord length. Three to five HPLC analyses were carried out on each cord length. The reproducibility of these HPLC analyses was within ± 5 percent.

TRANSFER LINE--FLSC

HNS-II was removed from approximately 2 cm of the FLSC line and the material was mechanically blended. Samples were weighed using a Mettler microbalance and were then dissolved in DMSO solvent. Three to five HPLC analyses were carried out on each cord length. The reproducibility of these HPLC analyses was within ± 3 percent.

PHOTODECOMPOSITION PRODUCTS

The photoproducts were removed from the surface of the irradiated solid HNS and HNBiB by solvent extraction. A somewhat preliminary separation was attained with subsequent extractions using these solvents added in the following sequence (1) ethyl ether, (2) benzene, (3) acetone, (4) methanol, and (5) water. The photolyzed material plus solvent was placed in an ultrasonic bath for approximately 15 minutes. Additional solvent was added until there was no further evidence of product solubility as determined by TLC. The compounds listed in the following tables were identified in a variety of ways. After separation by column, and/or preparative, thin-layer chromatography, characterization of the compounds was achieved with the aid of NMR, TLC, and HPLC analysis (see Tables 2, 3, 4, and Figure 5). Confirmation of the structures, when possible, was accomplished by comparison with authentic compounds.

TABLE 2. NMR DATA OF HNS AND HNB1B PHOTOLYSIS PRODUCTS

| Chemical Shift (Hz) in Solvents | | | | |
|---------------------------------|----------------|----------------|---------------------|----------------|
| <u>Compound*</u> | <u>DMSO</u> | <u>ACETONE</u> | <u>ACETONITRILE</u> | <u>BENZENE</u> |
| PiCHO | 1828 (s,2H)(a) | 1847 | 1862 | 1596 |
| | 2102 (s,1H) | 2117 | 2137 | 1960 |
| PiCH ₂ OH | 964 (s,2H) | 1003 | 1022 | 839 |
| | 1790 (s,2H) | 1773 | 1793 | 1530 |
| Anil | 1724 (d,1H) | 1740 | -- | 1502 |
| | 1862 (m,1H) | 1830 | -- | 1605 |
| | 2112 (d,1H) | 2055 | -- | 1711 |
| TNB | 1828 (s,2H) | 1851 | -- | 1648 |
| PiCOOH | 1822 (s,2H) | 1852 | -- | 1593 |
| TNT | 506 (s,3H) | 540 | -- | -- |
| | 1796 (s,2H) | 1805 | -- | -- |
| Iso-Anil | -- | 1792 (d,1H) | -- | -- |
| | -- | 1822 (m,1H) | -- | -- |
| | -- | 1930 (d,1H) | -- | -- |
| NODNBA | -- | 1547 (d,1H) | 1563 | 1325 |
| | -- | 1867 (d,1H) | 1878 | 1650 |
| PiCN | 1867 (s,2H) | --- | -- | 1595 |
| HNS | 1432 (s,2H) | --- | 1435 | -- |
| | 1814 (s,4H) | --- | 1820 | -- |
| HNB1B | 678 (s,4H) | --- | 714 | -- |
| | 1810 (s,4H) | --- | 1784 | -- |

(a) Spectra were determined on a Varian XL-200 Fourier Transform NMR spectrometer. Chemical shift data are reported in Hz downfield from TMS. The values in parentheses indicate multiplicity and integrated areas, respectively. s = singlet, d = doublet, m = multiplet.

* see Nomenclature

TABLE 3. TLC^(a) DATA OF THE IDENTIFIED HNS AND HNBIB PHOTOLYSIS PRODUCTS

| Compound* | R _f (benzene) |
|---------------------------|--------------------------|
| TNT | 0.86 |
| TNB | 0.77 |
| iso-anil | 0.73 |
| anil | 0.66 |
| HNBIB | 0.58 |
| PiCHO | 0.53 |
| PiCN | 0.45 |
| HNS | 0.38 |
| PiCH ₂ OH | 0.18 |
| dark brown tacky material | 0.00 |

| Compound | R _f (benzene:ether:ethanol) 50 : 30 : 20 |
|---------------------------|--|
| PiCOOH | 0.71 |
| dark brown tacky material | 0.00 - 0.35 |

(a) Thin layer plates prepared with Brinkman Silica gel - HF 254. Spot visualization by UV lamp. R_f taken from leading edge of spot.

* See Nomenclature

TABLE 4. HPLC DATA OF THE IDENTIFIED HNS AND HNBIB PHOTOPRODUCTS

| Compound* | Retention Time in Minutes (R_t) |
|----------------------|-------------------------------------|
| PICOOH | 3.35 |
| NODNBA | 3.63 |
| PICN | 7.10 |
| PICH ₂ OH | 7.65 |
| PICHO | 8.26 |
| TNB | 9.76 |
| iso-anil | 10.36 |
| anil | 10.95 |
| TNT | 12.03 |
| HNS | 13.01 |
| HNBIB | 18.41 |

HPLC Conditions:

Isocratic elution

Detector wavelength 254nm

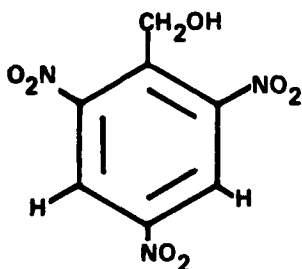
Cartridge - Radial-PAK A with RCM-100 Compression Module

Flow rate - 0.5 mL/minute

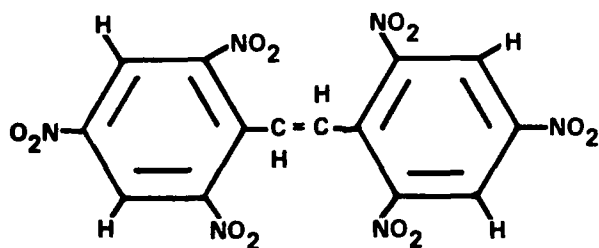
Mobile phase - 70% methanol/30% H₂O, (v,v)

Chart speed - 0.5 cm/minute

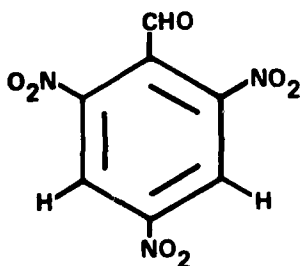
* See Nomenclature



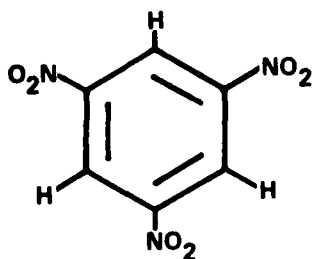
2,4,6 - TRINITROBENZYL ALCOHOL (PICH₂OH)
PHOTO PRODUCT OF HNBIB



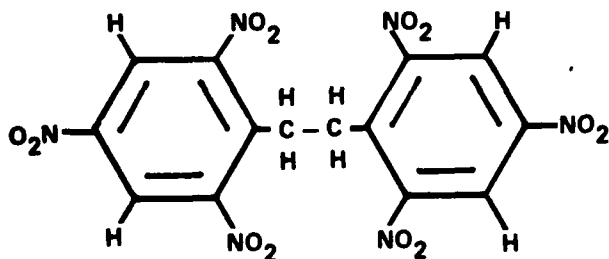
2,2',4,4',6,6' - HEXANITROSTILBENE (HNS)
PHOTO PRODUCT OF HNBIB



2,4,6 - TRINITROBENZALDEHYDE (PICHCHO)
PHOTO PRODUCT OF HNS AND HNBIB

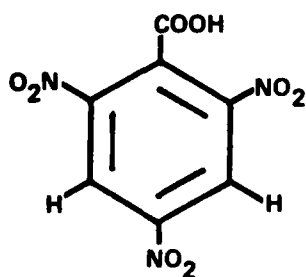


1,3,5 - TRINITROBENZENE (TNB)
PHOTO PRODUCT OF HNS AND HNBIB

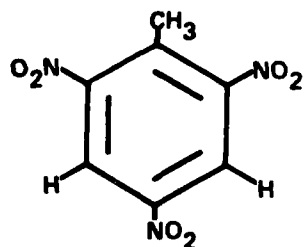


2,2',4,4',6,6' - HEXANITROBIBENZYL (HNBIB)
DIPICRYLETHANE (DPE)

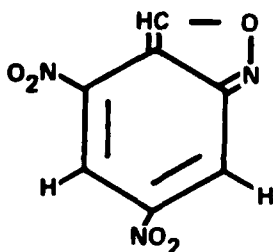
FIGURE 5. COMPOUNDS IDENTIFIED AS PHOTODECOMPOSITION PRODUCTS OF HNS AND HNBIB



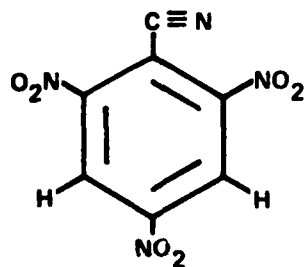
2,4,6 - TRINITROBENZOIC ACID (PicOOH)
PHOTO PRODUCT OF HNS AND HNBIB



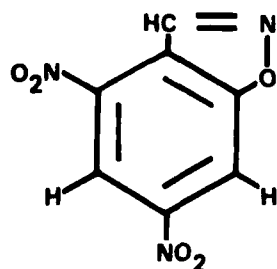
2,4,6 - TRINITROTOLUENE (TNT)
PHOTO PRODUCT OF HNBIB



4,6 - DINITRO [2,1] BENZISOXAZOLE
4,6 - DINITROANTHRANIL (ANIL)
PHOTO PRODUCT OF HNS AND HNBIB

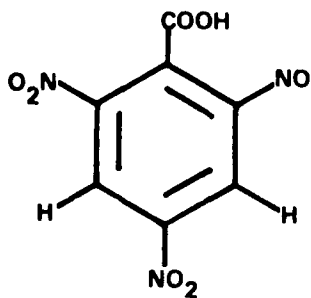


2,4,6 - TRINITROBENZONITRILE (PicN)
PHOTO PRODUCT OF HNBIB



4,6 - DINITROISOANTHRANIL
(iso - anil)
PHOTO PRODUCT OF ANIL

FIGURE 5. COMPOUNDS IDENTIFIED AS PHOTODECOMPOSITION PRODUCTS OF HNS AND HNBIB (Cont.)



2 - NITROSO - 4,6 - DINITROBENZOIC ACID
(NODNBA)
PHOTO PRODUCT OF PiCOOH

DARK BROWN TACKY MATERIAL

PHOTO PRODUCTS OF HNS AND HNBIB

WAXY OIL OR SOLID

PHOTO PRODUCT OF HNS AND HNBIB

FIGURE 5. COMPOUNDS IDENTIFIED AS PHOTODECOMPOSITION PRODUCTS
OF HNS AND HNBIB (Cont.)

CHAPTER 3

RESULTS AND DISCUSSION

PHASE I, II, AND III

HNS and HNBiB in solution and as solids become intensely colored when exposed to sunlight or room light. Solutions of HNS and HNBiB in organic solvents, such as acetonitrile, DMSO, and methanol, undergo a series of color changes (light yellow→pink→rose→gold→orange→orange brown) over a period of several hours exposure to light. The surface of the solid materials also discolors (from a light yellow→dark brown) after exposure to sunlight. As far as could be determined, the HNS photoproducts observed in both the solid or solution studies using either sunlight or the Hg source were similar, differing only in concentration. This same statement holds for the HNBiB photoproducts. Irradiated samples of HNS and HNBiB were found to contain numerous (~15) compounds. Identifying the initial photoproducts was further complicated because several of the primary photoproducts (PiCHO, anil, PiCH₂OH) were found to be more photo labile than the starting materials. The data indicate that photolyzed HNS and HNBiB result in a complex mixture of nitro bodies similar to (1) compounds observed during thermal degradation,⁶ (2) by-products⁷ isolated from the Shipp HNS synthesis process,⁸ (3) compounds isolated from irradiated aqueous solutions of TNT (pink water),⁹ and (4) compounds observed during the thermal degradation of TNT.¹⁰ The products isolated from irradiated HNS included: 2,4,6-trinitrobenzaldehyde (PiCHO); 4,6-dinitroanthranil (Anil); 1,3,5-trinitrobenzene (TNB); 2,4,6-trinitrobenzoic acid (PiCOOH); and 4,5-dinitroisoanthranil (iso-Anil). The products isolated from irradiated HNBiB include: PiCHO, Anil, TNB, PiCOOH, iso-Anil, TNT, trinitrobenzyl alcohol (PiCH₂OH), 2,4,5-trinitrobenzonitrile (PiCN), and HNS (see Figure 5).

At least six and possibly ten products observed early in the HNS and HNBiB photodecomposition defied separation and identification due to (1) the trace quantities that were formed and (2) the instability of the compounds as evidenced by TLC and HPLC. The majority of these photoproducts are considered to be relatively polar compounds since they (1) remained at the origin when chromatographed with the polar solvent mixture, benzene:ether:ethanol (50:30:20) v,v,v, and (2) eluted between 2 to 4 minutes on the HPLC using a (70:30) methanol:water v,v solvent mixture. On further irradiation, a dark brown residue, as well as a waxy-type substance, was observed. Properties of this relatively insoluble residue include:

1. dark brown, tacky material,
2. solubility in ether, benzene, acetonitrile, and water changes with time as evidenced by TLC and HPLC,

3. no separations observed using polar solvents with TLC and HPLC,
4. spraying the TLC tail with EDA/DMSO intensified the color, indicating the possible presence of additional nitroaromatic compounds.

The waxy oil and/or waxy solid substance contained high molecular weight and/or polar compounds, since most of this material (<70 percent) remained at the origin when chromatographed with 100 percent methanol. Attempts at differentiation (TLC and column chromatography) with a variety of solvents and support phases (silica gel, kieselgehr, and polyamide) failed to give any usable separations. The only stable photoproduct isolated was TNB which did not degrade even after 3 hours of irradiation at a distance of 2 cm from the Hg lamp. LC analysis after irradiation indicated no decomposition of the TNB.

Preliminary kinetic data on the photodecomposition of both HNS and HNBiB when exposed to solar radiation can be found in Tables 5 and 6. Of the two starting materials (HNS and HNBiB), the data indicate that HNBiB is approximately five times more photosensitive than the HNS. Several of the benzene soluble photoproducts (PiCHO, Anil, PiCH₂OH) were found to be more photolabile than either HNS or HNBiB.

PHASE IV

The silver-sheathed SMDC's and FLSC's, total length 160 to 180 mm, were fabricated at ET, Fairfield, California. ET's loading operators found the irradiated HNS-I and HNS-II materials less electrostatic, therefore, easier to load than the reference HNS-I and HNS-II. This included both the booster tips and the transfer lines.

PHASE V

The HNS-I and HNS-II obtained from the NASA/LBJ Space Center was used as the reference material for Phase V of this study. The time parameters arbitrarily selected for the direct sunlight irradiation were 4 hours, while irradiation by the Hg lamp in a pyrex cooling jacket was set at 30 minutes with the sample 3.5 cm from the source. These conditions are considered to be beyond those seen by HNS in normal synthesis and loading operations.

Chemical, photographic, and performance measurements were obtained on the three sample types: reference material, sunlight irradiated reference material, and Hg lamp irradiated reference material, which were fabricated into 2-1/2 grains/foot, silver-sheathed, SMDC lines and 20 grains/foot silver-sheathed FLSC lines. HPLC and performance data of the SMDC lines containing HNS (reference, sunlight-irradiated, Hg lamp-irradiated) can be found in Table 7. The Vacuum Thermal Stability (VTS)¹¹ data of the HNS can be found in Table 8. HPLC and performance data of the SMDC lines containing HNS (reference, sunlight, Hg lamp) exposed to 350°F for 50 hours can be found in Table 9. HPLC and performance data of the SMDC lines containing HNS (reference, sunlight irradiated, Hg lamp irradiated) exposed to 375°F for 50 hours can be found in Table 10. Due to the limited number (8) of samples tested for performance, no statistically significant conclusions could be drawn.

TABLE 5. HPLC ANALYSES OF SUNLIGHT IRRADIATED HNS AND HNBiB IN DMSO

(A) HPLC ANALYSES OF SUNLIGHT IRRADIATED HNS IN DMSO^(a)

| <u>Irradiation Time</u> <u>(in minutes)</u> | <u>% HNS</u> | <u>% Polar</u> <u>Compounds^(b)</u> | <u>% TNB</u> | <u>% PiCHO</u> | <u>% Anil</u> |
|--|--------------|--|--------------|----------------|---------------|
| 0 | 100.0 | 0.5 | 0 | 0 | 0 |
| 140 | 19.5 | 30.3 | 1 | 2 | 1 |
| 195 | 10.8 | 37.1 | 2 | 3 | 3 |
| 255 | 5.6 | 39.2 | 3 | - | 3 |
| 320 | 3.5 | 43.0 | 3 | 1 | 2 |

(B) HPLC ANALYSES OF SUNLIGHT IRRADIATED HNBiB IN DMSO^(a)

| <u>Irradiation Time</u> <u>(in minutes)</u> | <u>% HNS</u> | <u>% HNBiB</u> | <u>% Polar</u> <u>Compounds^(b)</u> | <u>% TNT</u> | <u>%TNB</u> | <u>% PiCHO</u> | <u>% Anil</u> |
|--|--------------|----------------|--|--------------|-------------|----------------|---------------|
| 0 | 1.3 | 98.5 | 0.2 | 0 | 0 | 0 | 0 |
| 110 | 20.3 | 5.8 | 29.4 | 2 | - | 2 | 3 |
| 170 | 13.1 | 1.5 | 38.2 | 3 | 3 | 4 | 3 |
| 230 | 9.6 | 0.8 | 34.9 | 1 | 2 | 4 | 1 |
| 290 | 6.0 | 0.2 | 32.2 | 1 | 3 | 1 | 1 |

HNS $T_{1/2}$ = 80 minutes at ambient temperature

HNBiB $T_{1/2}$ = 52 minutes at ambient temperature

(a) Concentration of HNS and HNBiB in DMSO = 100ppm

(b) the percent polar material is determined from the integrated peak area of 2-4 minutes assuming the same response factor as HNS.

TABLE 6. HPLC ANALYSES OF SUNLIGHT IRRADIATED HNS AND HNBIB IN ACETONITRILE

(A) HPLC ANALYSES OF SUNLIGHT* IRRADIATED HNS IN ACETONITRILE^(a)

| <u>Irradiation Time (in minutes)</u> | <u>% HNS</u> | <u>% Polar Compounds^(b)</u> | <u>% TNB</u> | <u>% PiCHO</u> |
|--|--------------|--|--------------|----------------|
| 0 | 100.0 | 0.0 | 0 | 0 |
| 60 | 93.0 | 7.0 | 1 | 3 |
| 120 | 72.0 | 20.1 | 2 | 7 |
| 180 | 50.0 | 42.3 | 3 | 2 |
| 240 | 34.4 | 45.7 | 7 | 2 |
| 300 | 24.7 | 47.4 | 7 | 2 |
| 360 | 10.0 | -- | 7 | -- |

(B) HPLC ANALYSES OF SUNLIGHT* IRRADIATED HNBIB IN ACETONITRILE^(a)

| <u>Irradiation Time (in minutes)</u> | <u>% HNS</u> | <u>% HNBIB</u> | <u>% Polar Compounds^(b)</u> | <u>% TNB</u> | <u>% Anil</u> |
|--|--------------|----------------|--|--------------|---------------|
| 0 | 1.3 | 98.5 | 0.3 | 0 | 0 |
| 60 | 3.1 | 83.8 | 9.3 | - | 1 |
| 120 | 7.5 | 51.7 | 20.5 | 1 | 3 |
| 180 | 13.2 | 20.1 | 31.0 | 3 | - |
| 240 | 9.4 | 6.5 | 35.7 | 7 | - |
| 300 | 3.3 | 4.0 | 33.5 | 3 | 1 |

*Sunlight - thin cloud cover

HNS $T_{1/2}$ - 180 minutes at ambient temperatureHNBIB $T_{1/2}$ - 125 minutes at ambient temperature

(a) concentration of HNS and HNBIB in acetonitrile - 10ppm

(b) the percent polar material is determined from the integrated peak area of 2-4 minutes assuming the same response factor as HNS.

TABLE 7. HPLC AND PERFORMANCE DATA OF IRRADIATED HNS

| <u>Sample</u> | | <u>% HNS</u> | <u>% HNBiB</u> | <u>Av. Detonation Velocity (mm/sec)</u> | <u>Output Measurement Av. Dent (mils)</u> |
|---------------------------------|----|--------------|----------------|---|---|
| SMDC | | | | | |
| Reference ^(a) | BT | 98.1 | 0.5 | 6.28 | 0.013 |
| | TL | 97.8 | 0.3 | | |
| Sunlight | BT | 98.1 | 0.4 | 6.26 | 0.013 |
| | TL | 97.3 | 0.2 | | |
| Hg Lamp | BT | 98.7 | 0.5 | 6.44 | 0.014 |
| | TL | 97.7 | 0.3 | | |
| FLSC | | | | | |
| Reference ^(a) | TL | 99.6 | 0.3 | 7.05 | -- |
| Sunlight | TL | 100.0 | 0.3 | 7.03 | -- |
| Hg Lamp | TL | 99.5 | 0.3 | 7.05 | -- |
| -- not measured | | | | | |
| ^(a) Unphotolyzed HNS | | | | | |

TABLE 8. VACUUM THERMAL STABILITY (VTS)* DATA OF HNS SAMPLES

| | | cc/g corrected | | |
|--------------------------|----|-----------------|------------------------------------|-----------------------------------|
| | | 20 min Surge | 2 hour period (Excluded 20 min) | 2 hour period (Include 20 min) |
| <u>Sample</u> | | | | |
| SMDC | | | | |
| Reference ^(a) | BT | 1.48 | 1.12 | 2.60 |
| | TL | 0.603 | 0.644 | 1.25 |
| Sunlight | BT | 1.49 | 1.18 | 2.67 |
| | TL | 0.704 | 0.746 | 1.45 |
| Hg Lamp | BT | 1.85 | 1.15 | 3.00 |
| | TL | 0.700 | 0.707 | 1.41 |

* VTS testing at 260°C using procedure outlined in Simmons, Herbert T., Sr.,
 "Vacuum Thermal Stability Testing for Explosives," NOLTR 70-142,
 28 Oct 1970.¹¹

(a) Unphotolyzed HNS

The SEM data can be found in Figures 6 through 11 and the color macrophotographic data in Figures 12 through 17.

The average detonation velocity of the sunlight irradiated samples with additional thermal exposure to 350°F/50 hours decreased by approximately 3 percent when compared to the sunlight irradiated zero time (T_0) sample. All SMDC lines that were performance tested initiated along their entire length. With additional thermal input of 375°F/50 hours, all the SMDC lines failed to fully initiate. The failures were randomly distributed along the lines. None of the failures occurred in the booster tips. HPLC and performance data of the SMDC lines containing HNS (reference, sunlight, Hg lamp) exposed to 350°F for 50 hours can be found in Table 9.

The Hg lamp irradiated SMDC lines exposed to 350 degrees for 50 hours also exhibited a slight decrease in performance when compared to the Hg lamp irradiated zero time (T_0) sample. The average sample detonation velocity decreased from 6.44 mm/ μ sec) to 6.20 mm/ μ sec (4 percent). At additional thermal exposure to 375°F for 50 hours, all the SMDC lines failed to initiate. These failures also occurred randomly along the lines and did not occur in the booster tips.

The initial dark color of the FLSC reference sample (Figure 18, T_0) is attributed to the "stress relieve techniques"¹² used by ET. Additional thermal exposure (350°F, 375°F) of the FLSC lines appeared to have a minimal effect on their performance. The data (Tables 8, 9, and 10) show that the detonation velocity of the FLSC lines were not affected by either thermal exposure (350°F, 375°F) or irradiation (sunlight, Hg lamp).

The photographic data obtained on the FLSC samples can be found in Figures 18 through 20 and the SEM data in Figures 21 through 23.

TABLE 9. HPLC AND PERFORMANCE DATA OF IRRADIATED HNS EXPOSED TO 350°F FOR 50 HOURS

| <u>Sample</u> | | <u>% HNS</u> | <u>% HNBiB</u> | <u>Av. Detonation Velocity (mm/sec)</u> | <u>Output Measurement Av. Dent (mils)</u> |
|--|----|--------------|----------------|---|---|
| SMDC | | | | | |
| Reference ^(a) | BT | 99.0 | 0.4 | 6.06 | 0.014 |
| | TL | 97.2 | 0.2 | | |
| Sunlight | BT | 97.9 | 0.4 | 6.10 | 0.012 |
| | TL | 98.3 | 0.3 | | |
| Hg Lamp | BT | 98.0 | 0.4 | 6.20 | 0.013 |
| | TL | 97.8 | 0.2 | | |
| FLSC | | | | | |
| Reference ^(a) | TL | 98.7 | 0.3 | 7.05 | -- |
| Sunlight | TL | 99.7 | 0.4 | 7.03 | -- |
| Hg Lamp | TL | 98.7 | 0.3 | 7.05 | -- |
| -- not measured | | | | | |
| ^(a) Unphotolyzed HNS material | | | | | |

TABLE 10. HPLC AND PERFORMANCE DATA OF IRRADIATED HNS EXPOSED TO 375°F FOR 50 HOURS

| <u>Sample</u> | | <u>% HNS</u> | <u>% HNBiB</u> | <u>Av. Detonation Velocity (mm/sec)</u> | <u>Output Measurement Av. Dent (mils)</u> |
|--------------------------|----|--------------|----------------|---|---|
| SMDC | | | | | |
| Reference ^(a) | BT | 97.3 | 0.5 | 6.05 | 0.013 |
| | TL | 98.0 | 0.2 | | |
| Sunlight | BT | 98.3 | 0.5 | * | * |
| | TL | 98.2 | 0.3 | | |
| Hg Lamp | BT | 97.1 | 0.4 | * | * |
| | TL | 98.0 | 0.2 | | |

FLSC

| | | | | | |
|--------------------------|----|------|-----|------|----|
| Reference ^(a) | TL | 98.5 | 0.2 | 7.18 | -- |
| Sunlight | TL | 97.0 | 0.3 | 7.14 | -- |
| Hg Lamp | TL | 97.5 | 0.3 | 7.05 | -- |

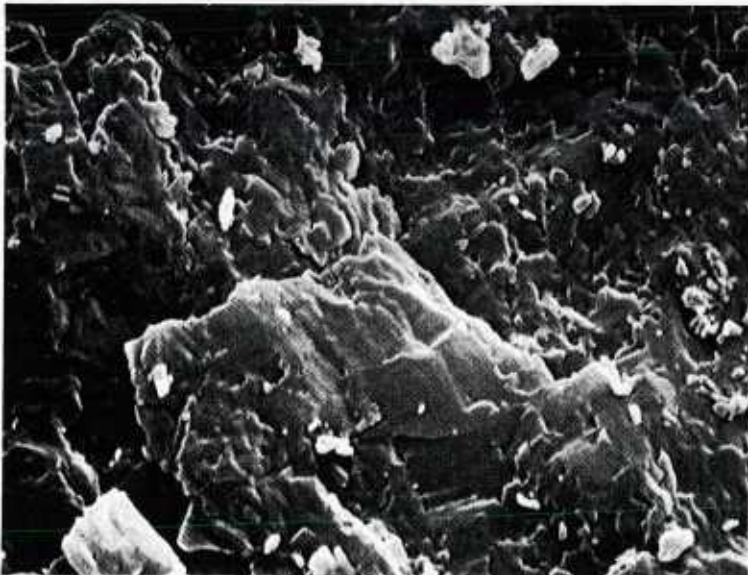
* lines did not fully initiate along the entire length

-- not measured

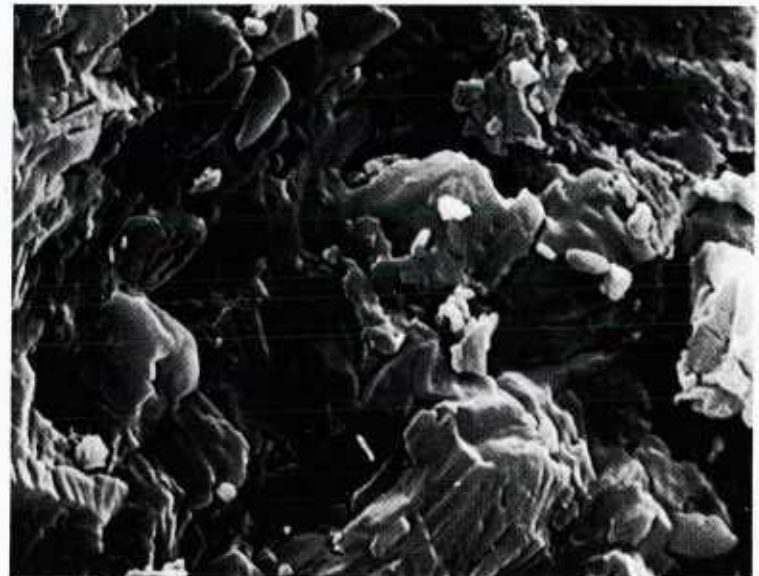
(a) Unphotolyzed HNS material



REFERENCE SAMPLE
(T₀)



350°F/50 HOURS

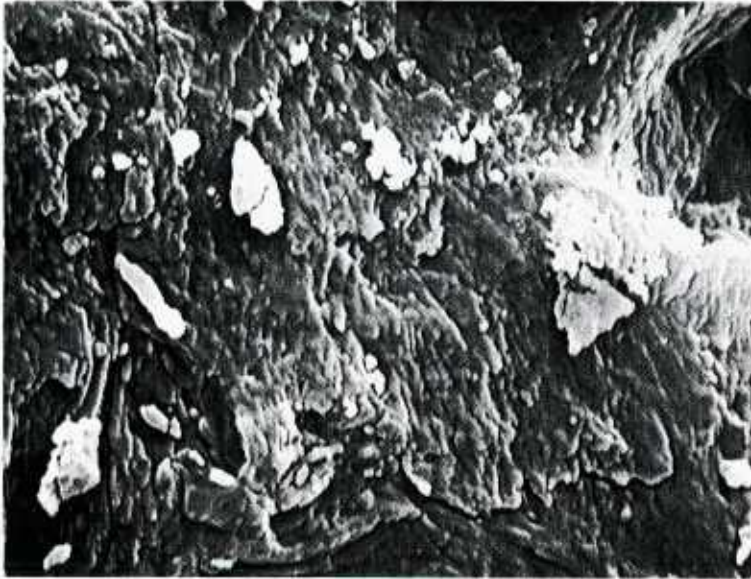


375°F/50 HOURS

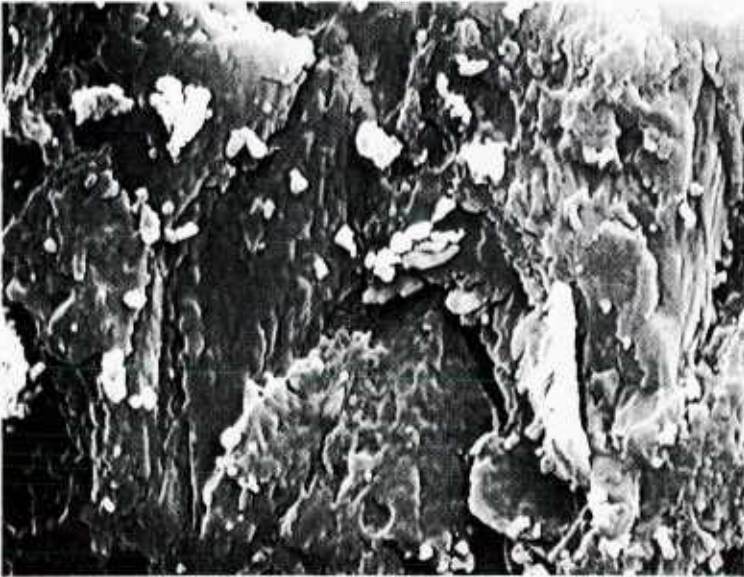
MAGNIFICATION 5000x

NSWC TR 90-60

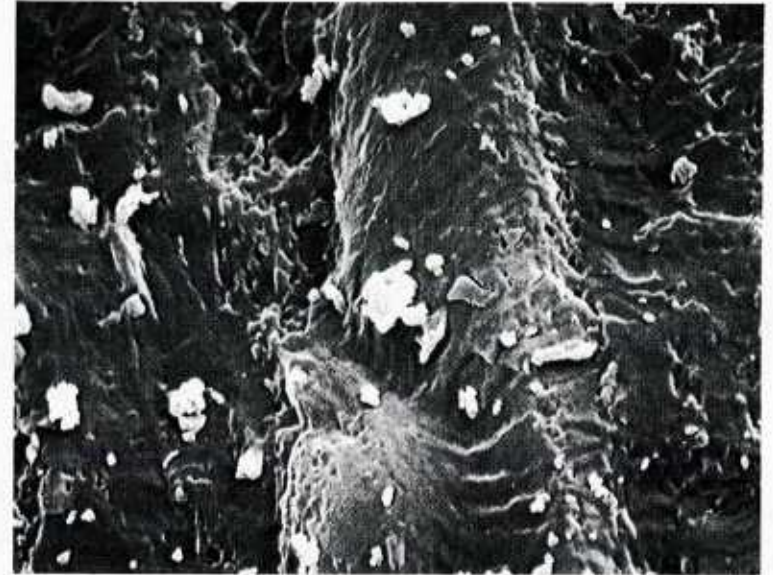
FIGURE 6. SCANNING ELECTRON MICROGRAPHS SMDC BOOSTER TIPS



REFERENCE SAMPLE
(T₀)



350°F/50 HOURS



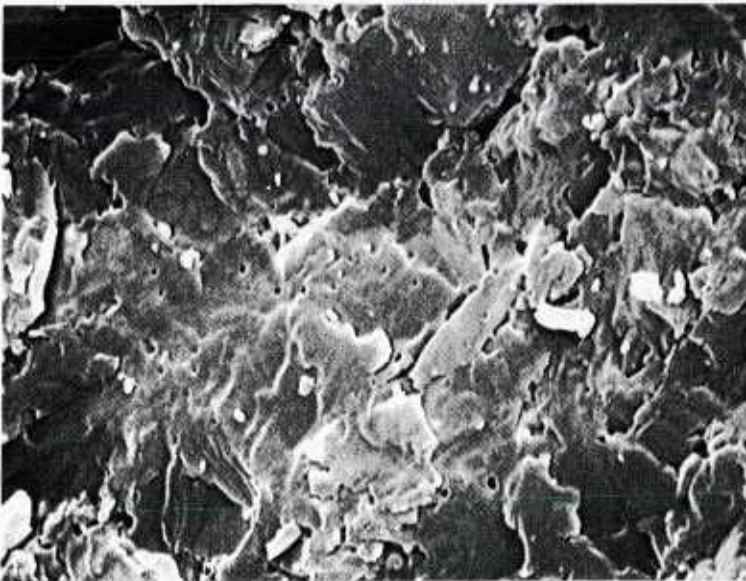
375°F/50 HOURS

MAGNIFICATION 5000x

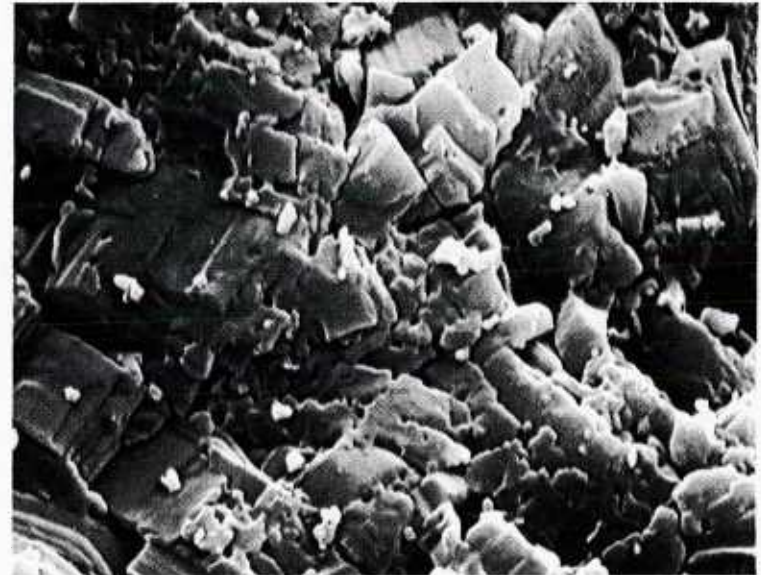
FIGURE 7. SCANNING ELECTRON MICROGRAPHS SMDC TRANSFER LINES



REFERENCE SAMPLE (T_0)



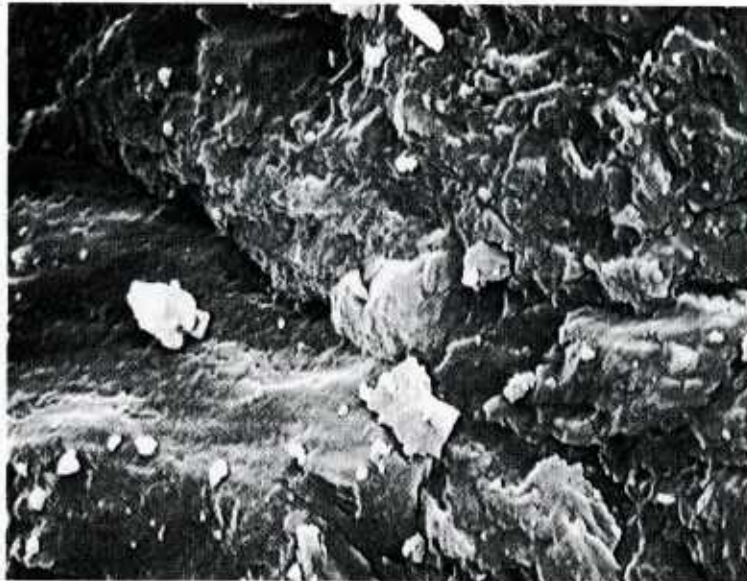
350°F/50 HOURS



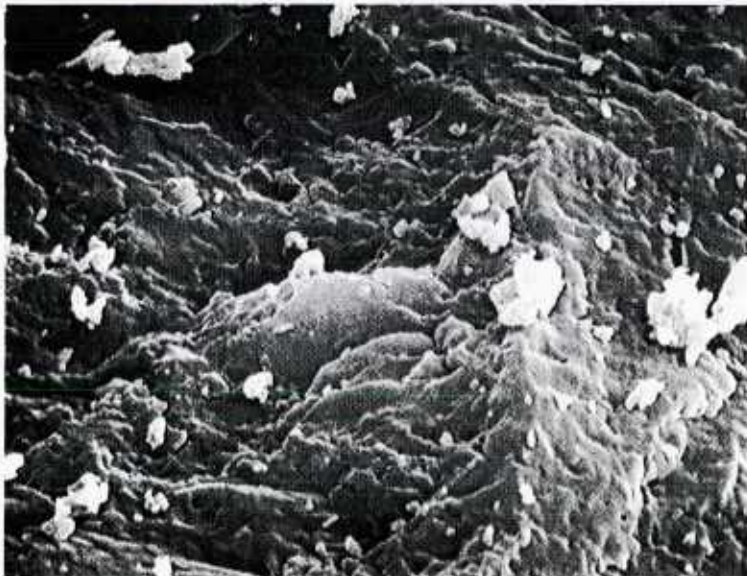
375°F/50 HOURS

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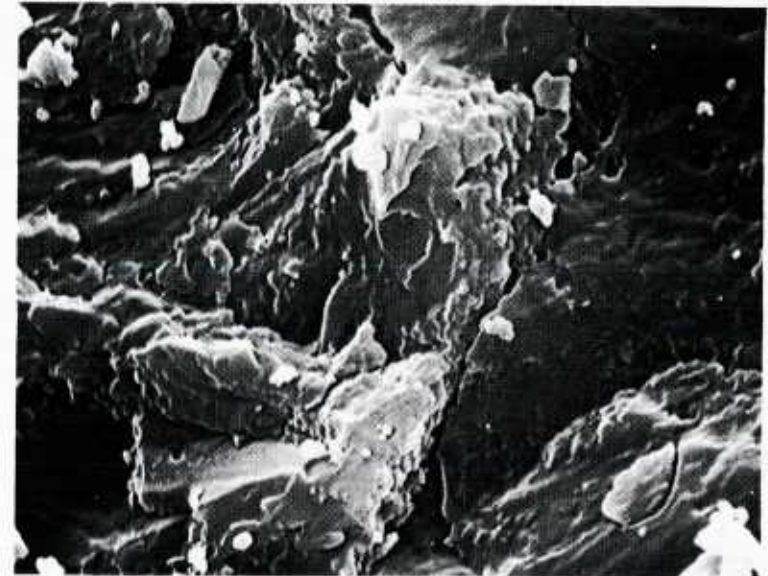
FIGURE 8. SCANNING ELECTRON MICROGRAPHS SMDC BOOSTER TIPS--SUNLIGHT IRRADIATION



REFERENCE SAMPLE (T_0)



350°F/50 HOURS



375°F/50 HOURS

MAGNIFICATION 5000x

NSWC TR 90-60

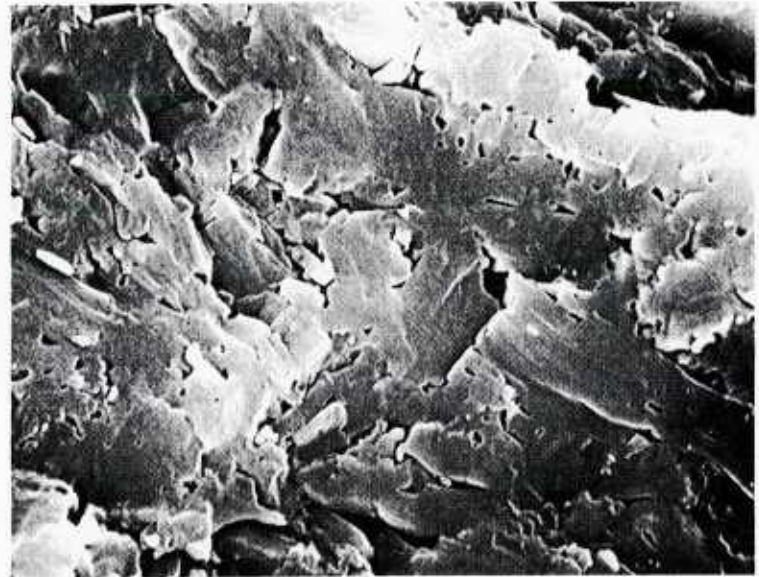
FIGURE 9. SCANNING ELECTRON MICROGRAPHS SMDc TRANSFER LINES--SUNLIGHT IRRADIATION



REFERENCE SAMPLE (T₀)



350°F/50 HOURS



375°F/50 HOURS

MAGNIFICATION 5000x

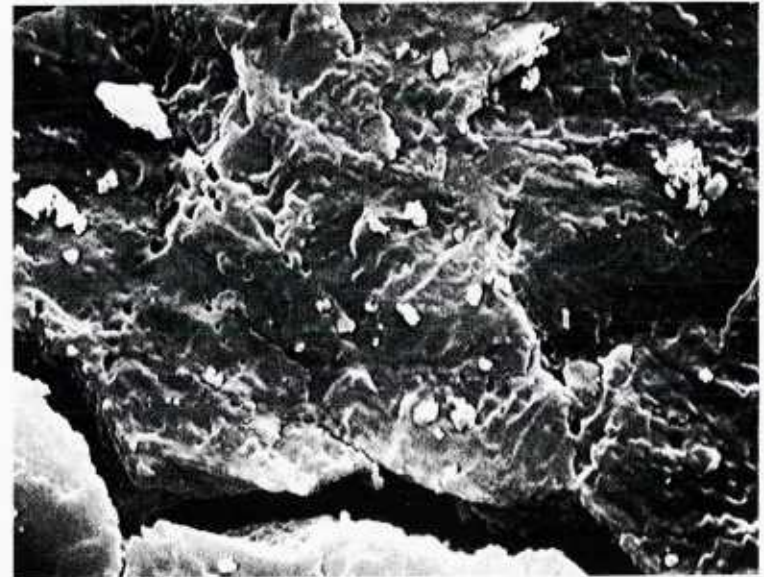
FIGURE 10. SCANNING ELECTRON MICROGRAPHS SMDC BOOSTER TIPS--Hg LAMP IRRADIATION



REFERENCE SAMPLE (T₀)



350°F/50 HOURS



375°F/50 HOURS

MAGNIFICATION 5000x

FIGURE 11. SCANNING ELECTRON MICROGRAPHS SMD C TRANSFER LINES--Hg LAMP IRRADIATION

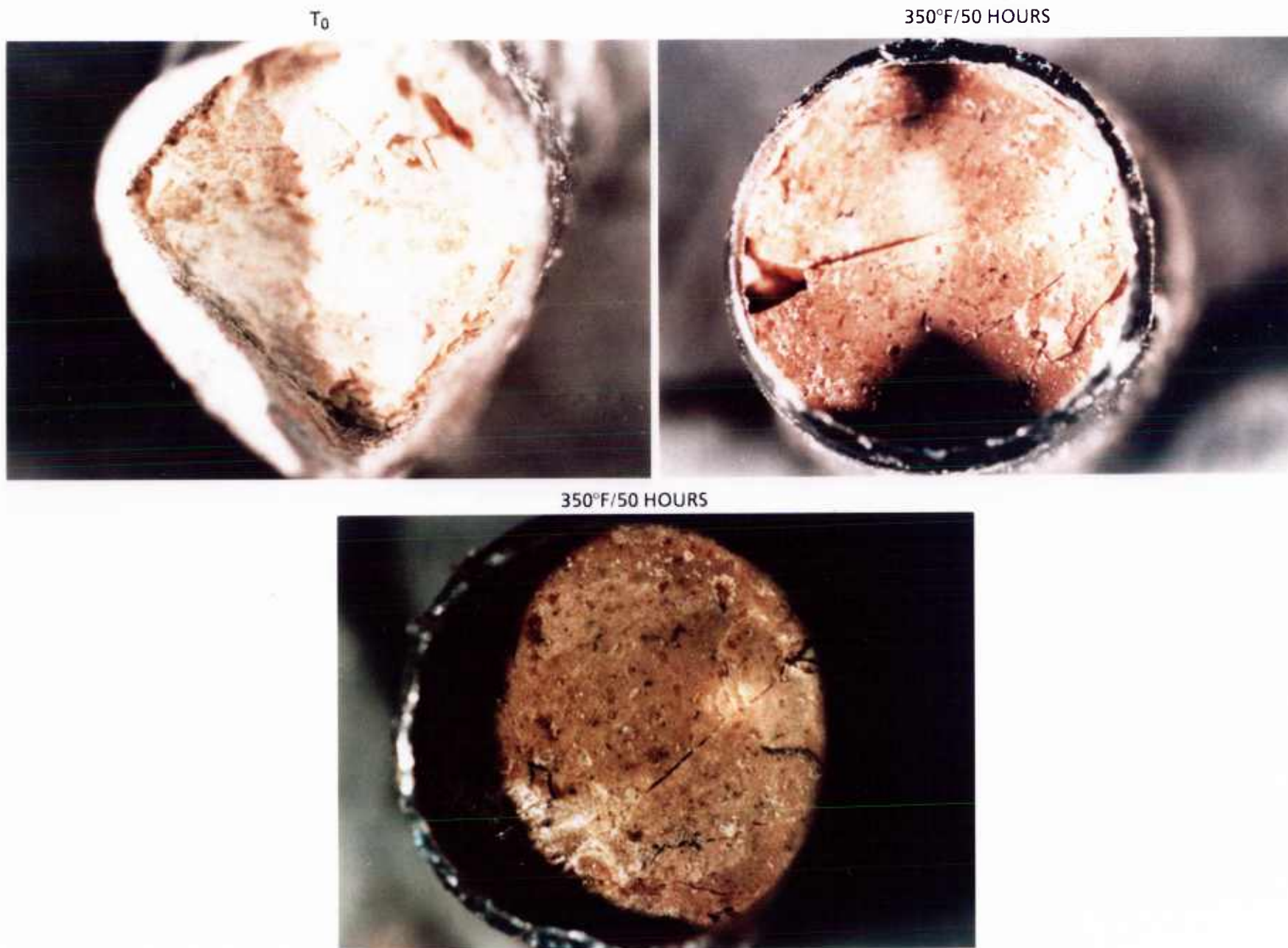
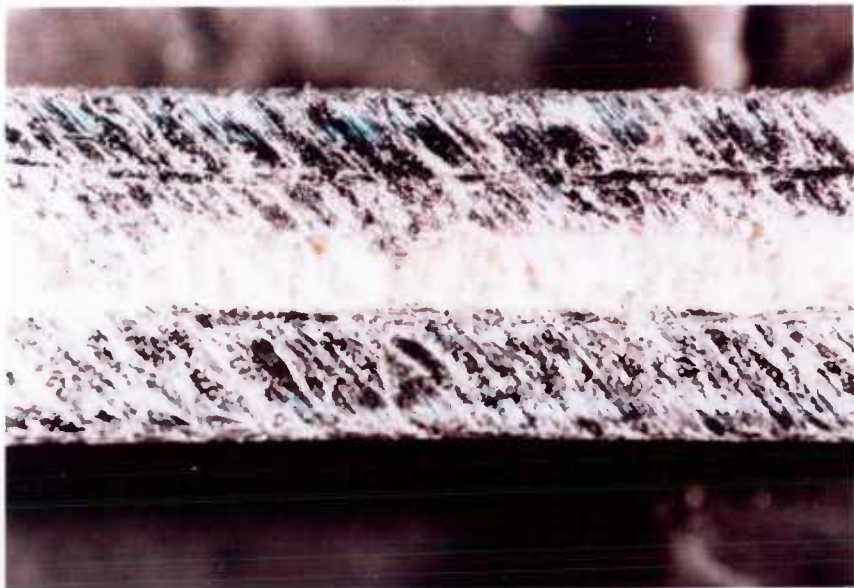
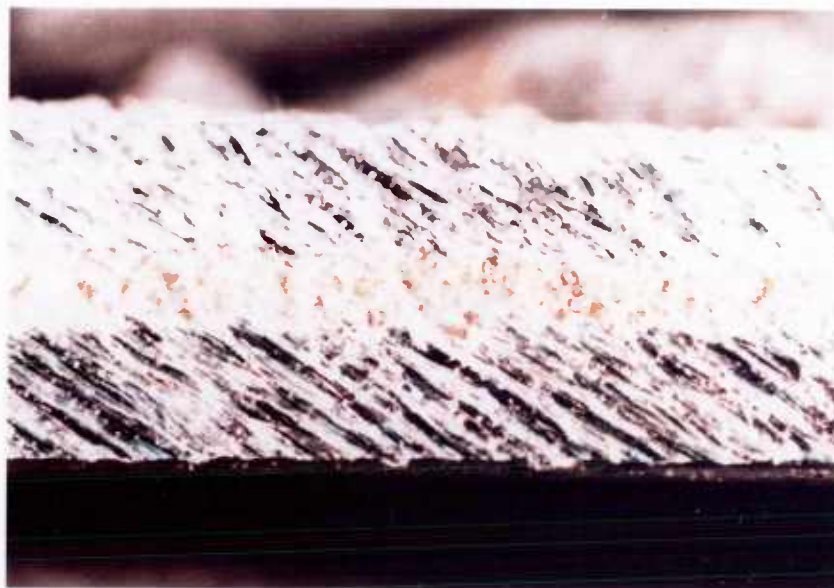


FIGURE 12. COLOR MACROPHOTOGRAPHS SMDC BOOSTER TIPS

T₀



350°F/50 HOURS



375°F/50 HOURS

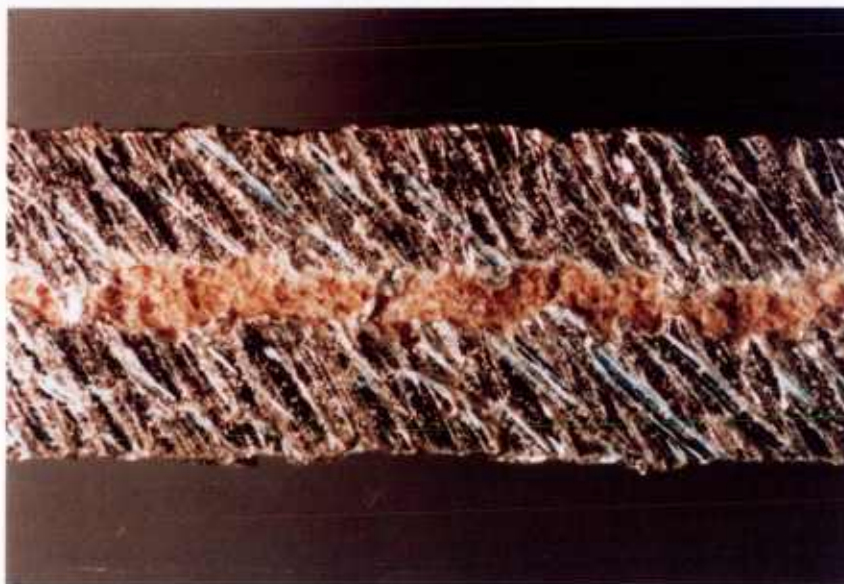


FIGURE 13. COLOR MACROPHOTOGRAPHS SMDC TRANSFER LINES



FIGURE 14. COLOR MACROPHOTOGRAPHS SMDC BOOSTER TIPS--SUNLIGHT IRRADIATION

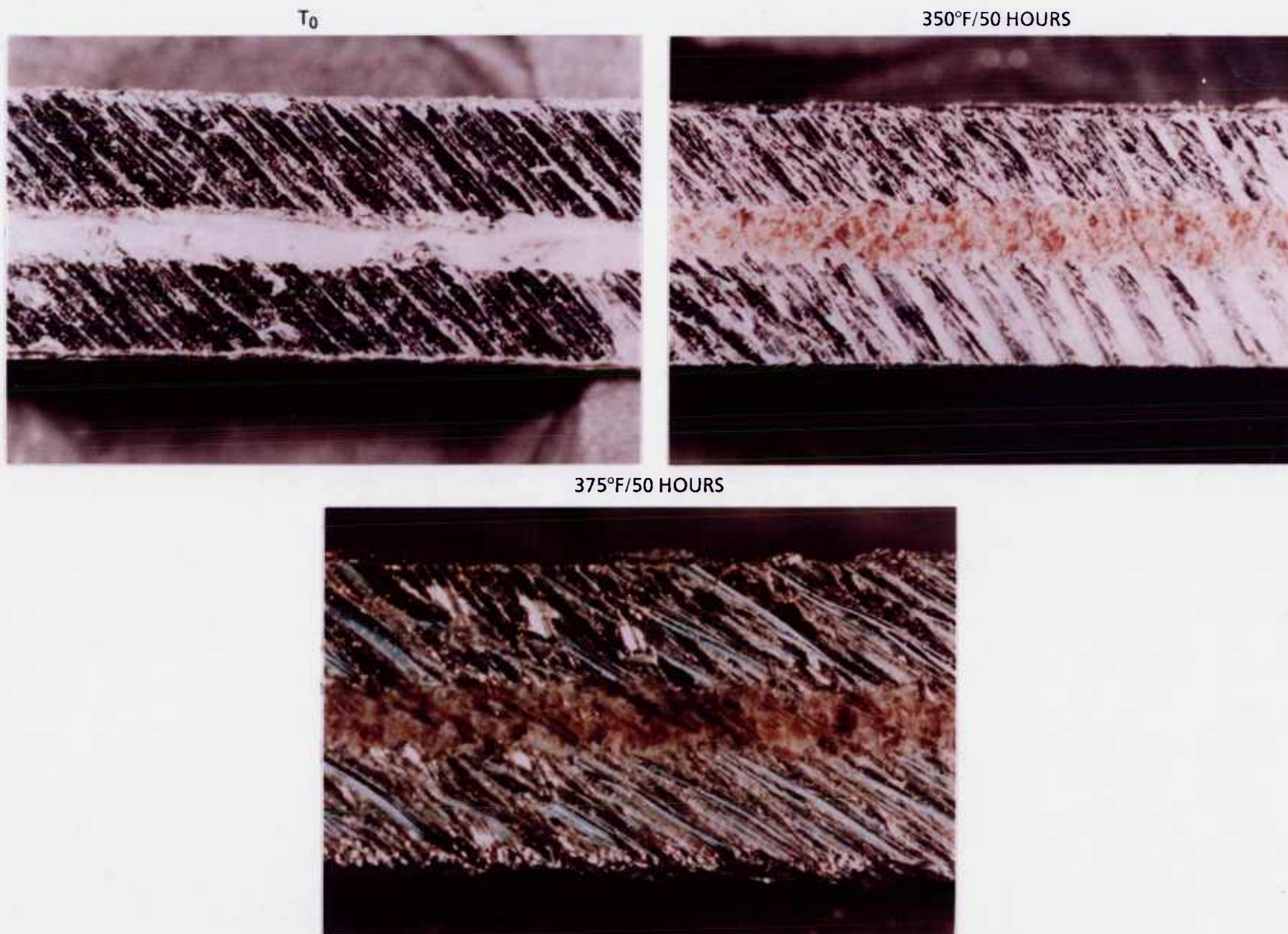
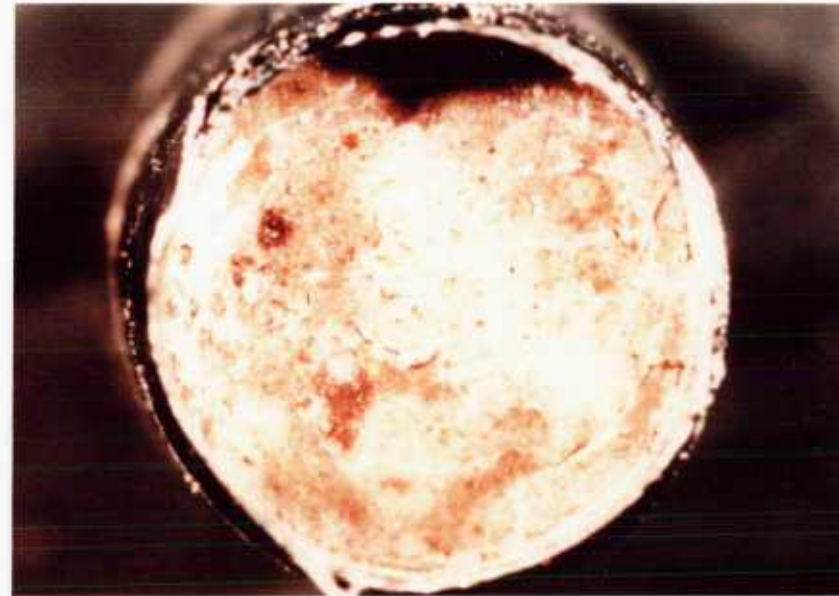


FIGURE 15. COLOR MACROPHOTOGRAPHS SMDC TRANSFER LINES--SUNLIGHT IRRADIATION

T₀

350°F/50 HOURS



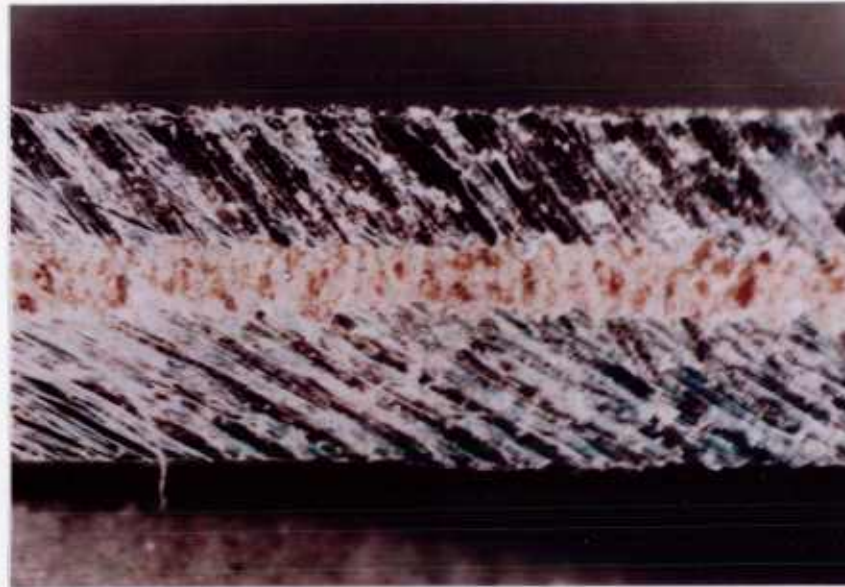
375°F/50 HOURS



FIGURE 16. COLOR MACROPHOTOGRAPHS SMDC BOOSTER TIPS--Hg LAMP IRRADIATION

T_0 

350°F/50 HOURS



375°F/50 HOURS

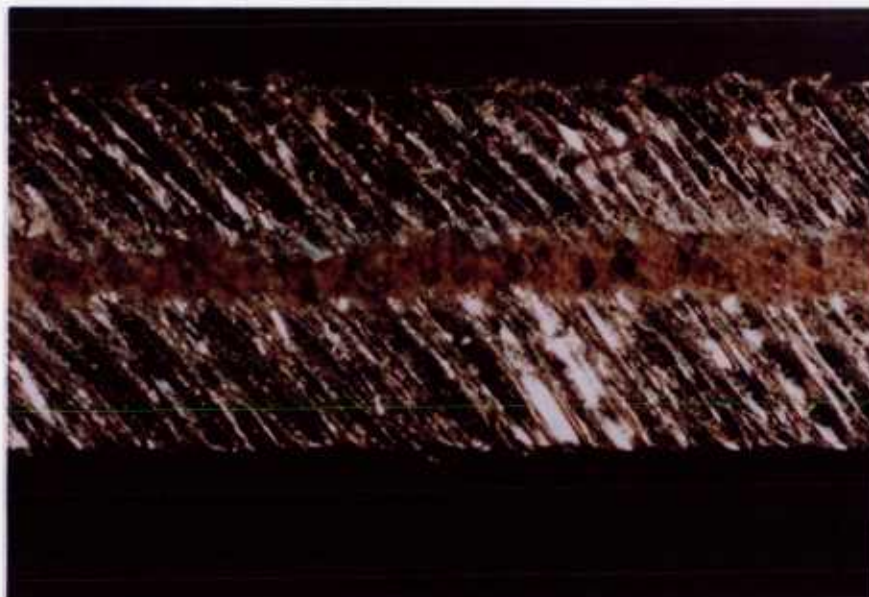


FIGURE 17. COLOR MACROPHOTOGRAPHS SMDC TRANSFER LINES--Hg LAMP IRRADIATION

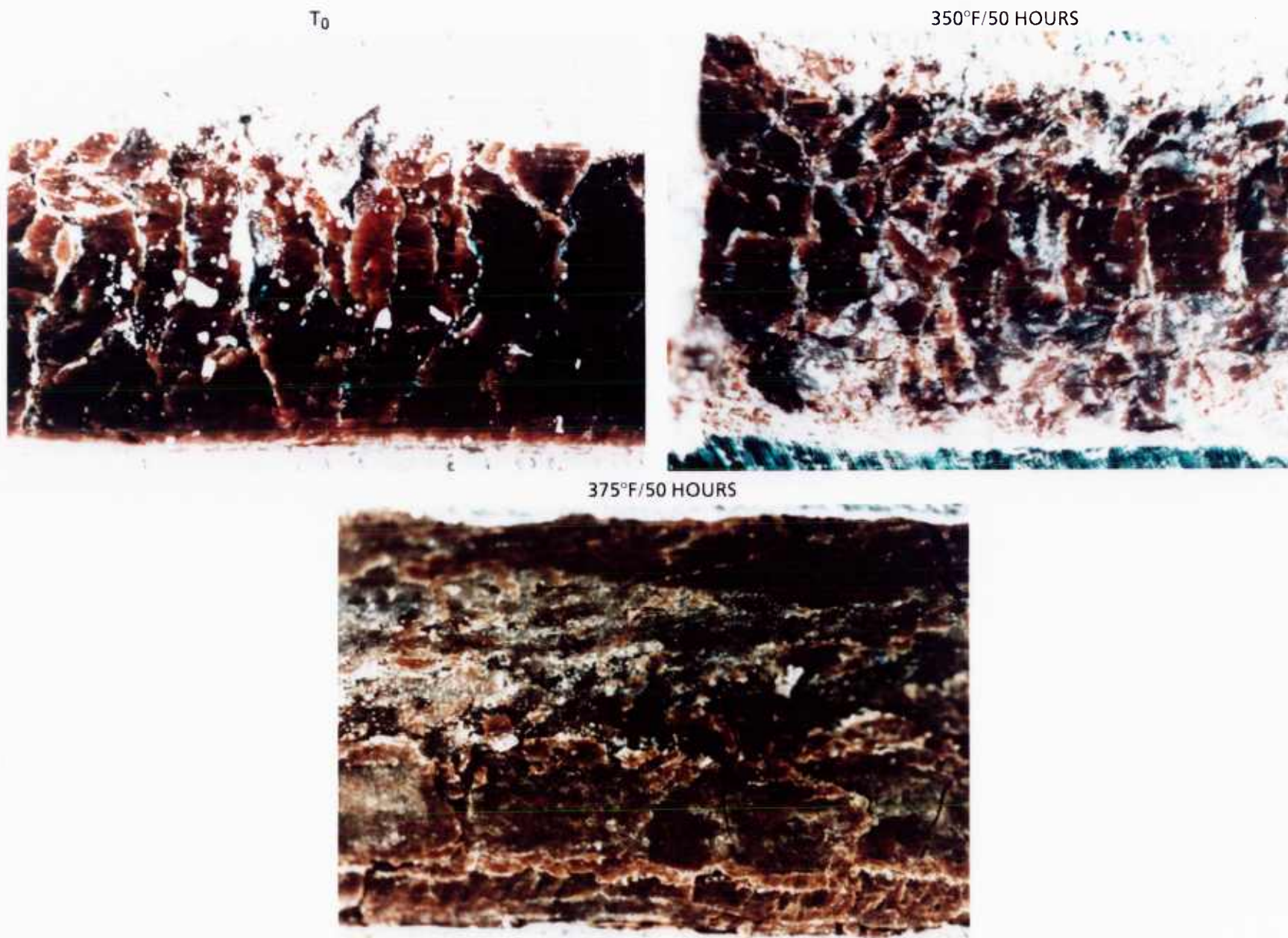


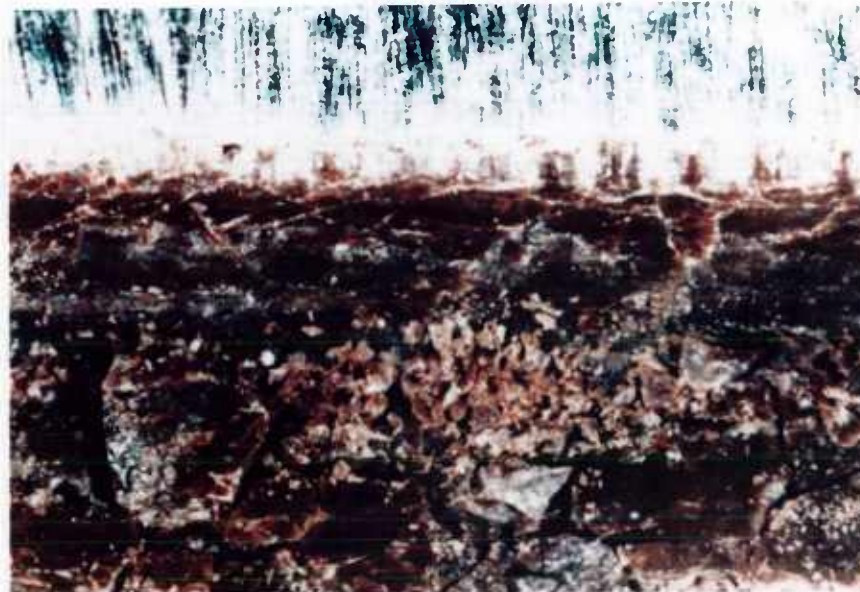
FIGURE 18. COLOR MACROPHOTOGRAPHS FLSC--REFERENCE SAMPLE

05/67

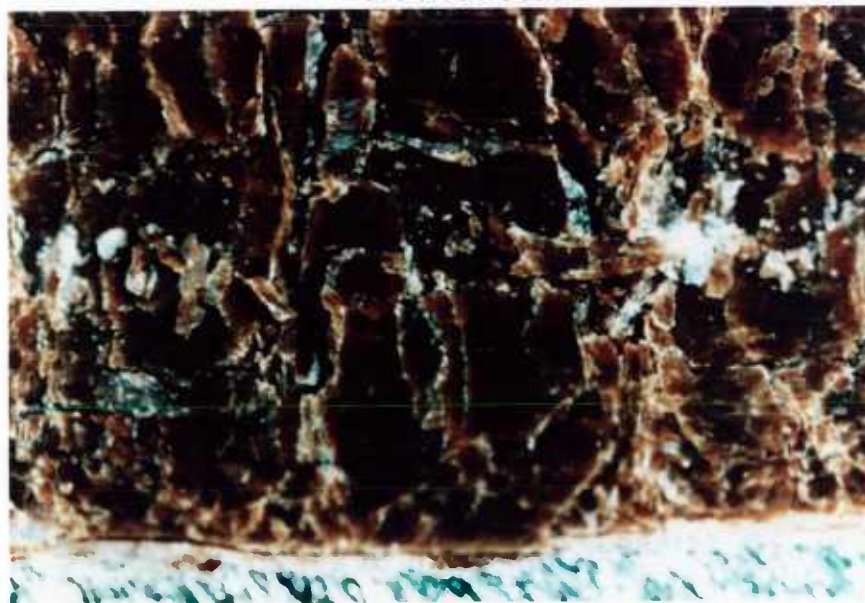
T₀



350°F/50 HOURS



375°F/50 HOURS



NSWC TR 90-60

FIGURE 19. COLOR MACROPHOTOGRAPHS FLSC--SUNLIGHT IRRADIATION

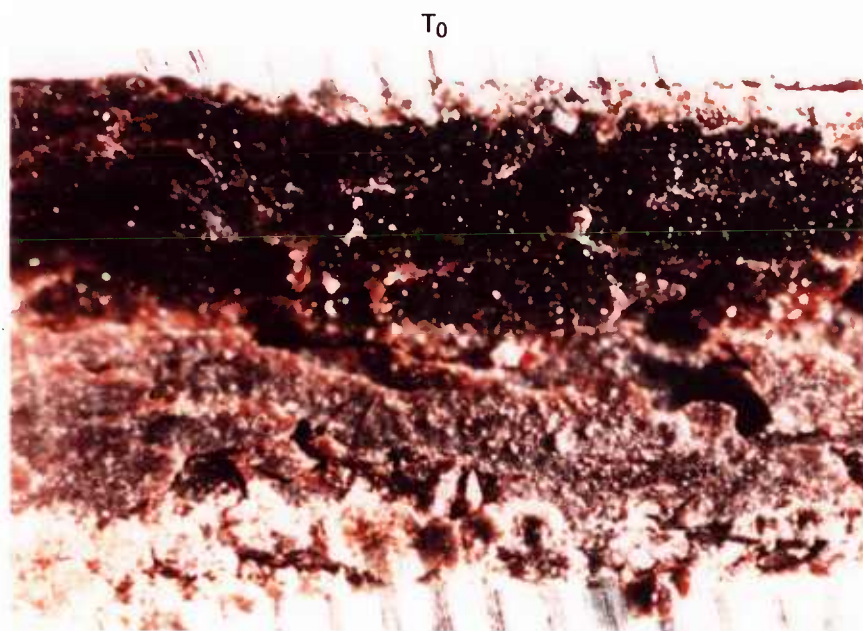
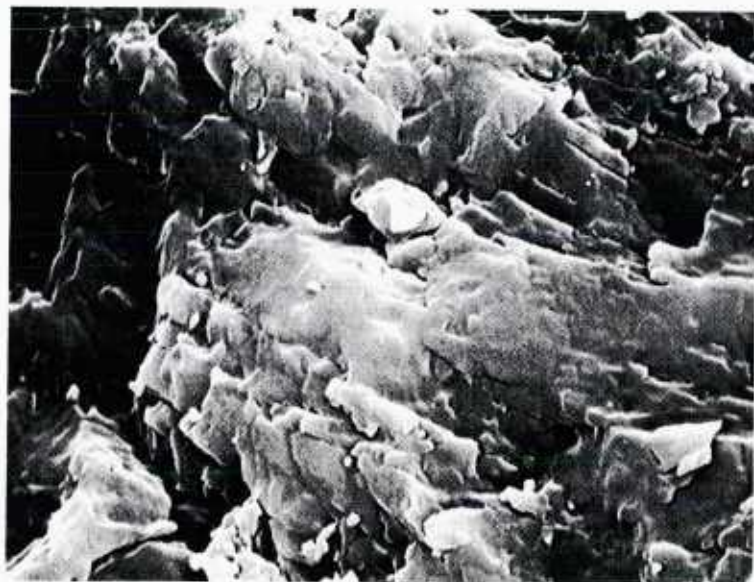
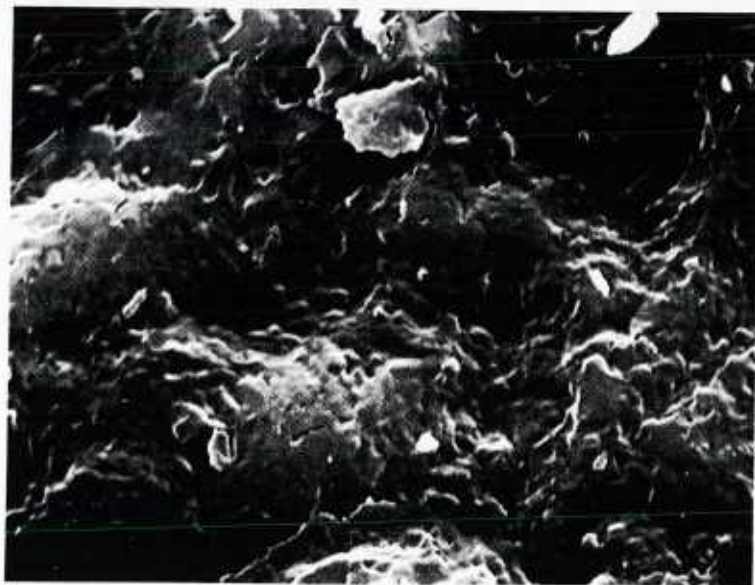


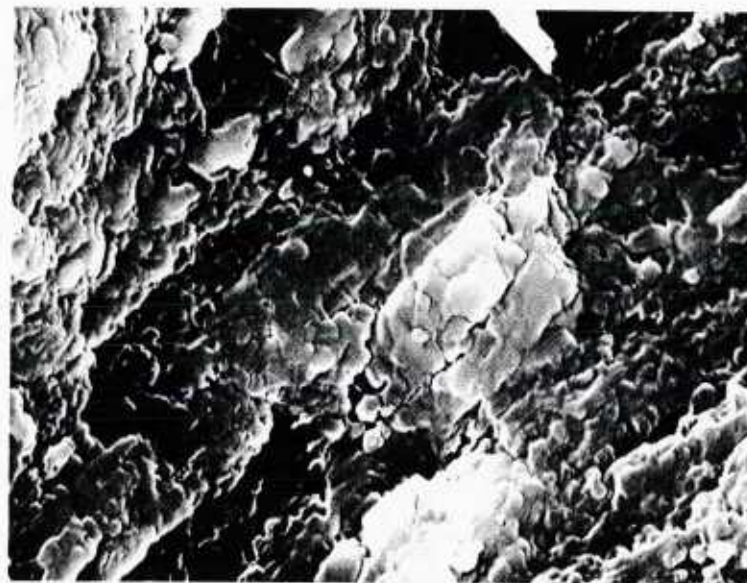
FIGURE 20. COLOR MACROPHOTOGRAPHS FLSC--Hg LAMP IRRADIATION



REFERENCE SAMPLE (T_0)



350°F/50 HOURS

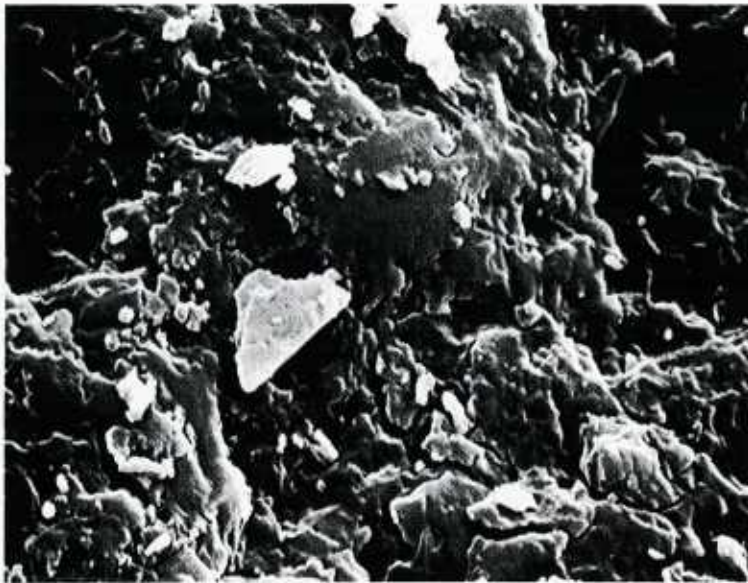


375°F/50 HOURS

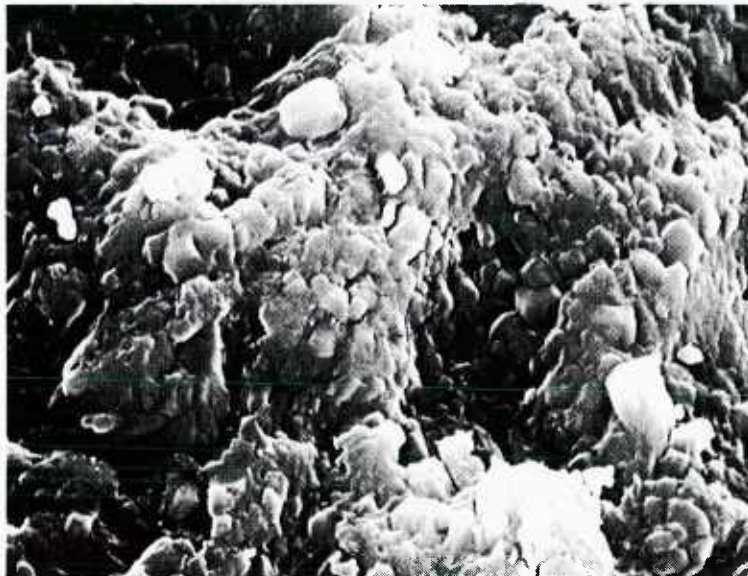
MAGNIFICATION 5000x

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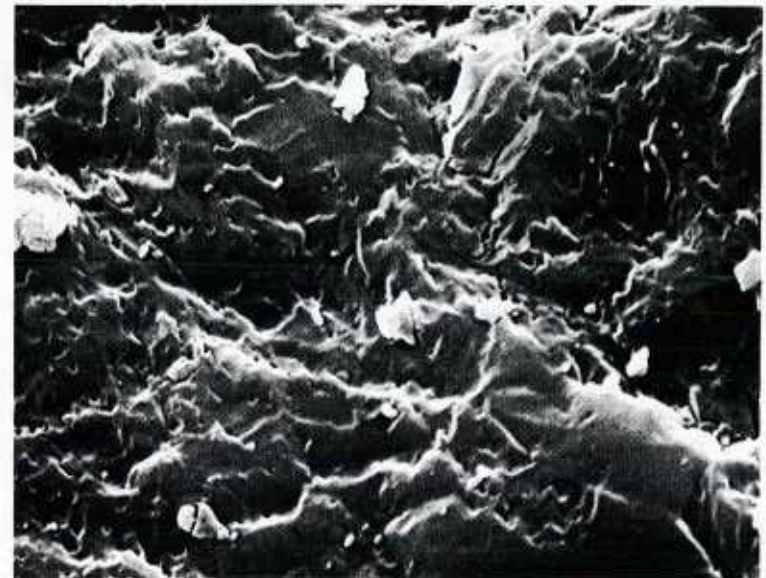
FIGURE 21. SCANNING ELECTRON MICROGRAPHS FLSC



REFERENCE SAMPLE (T_0)



350°F/50 HOURS

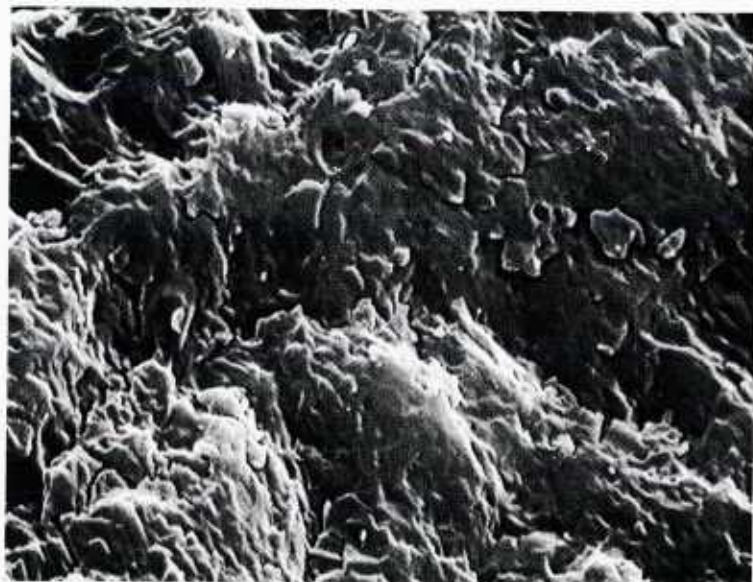


375°F/50 HOURS

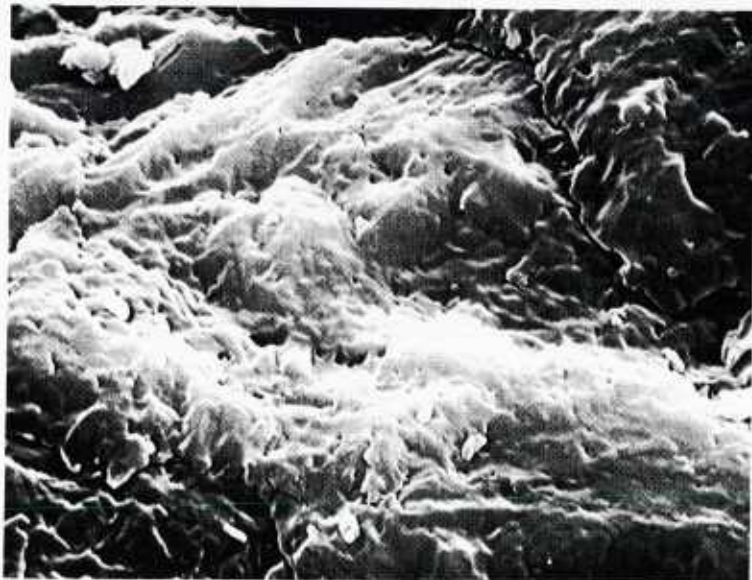
MAGNIFICATION 5000x

NSWC TR 90-60

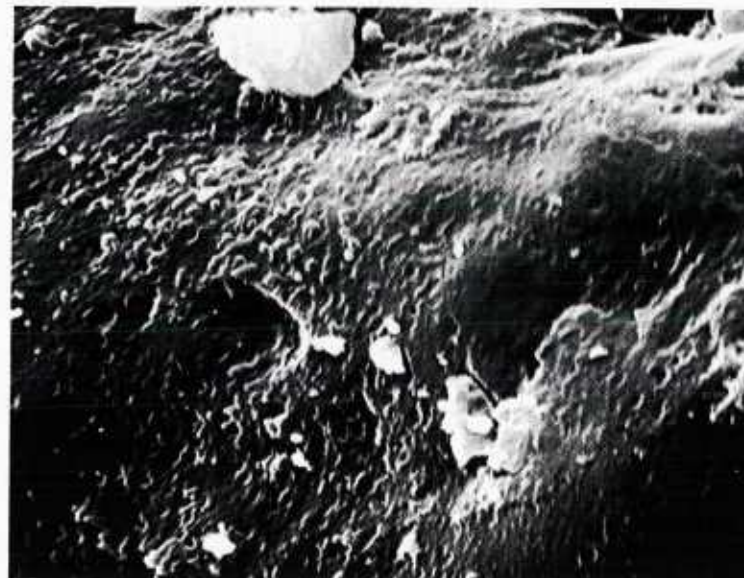
FIGURE 22. SCANNING ELECTRON MICROGRAPHS FLSC--SUNLIGHT IRRADIATION



REFERENCE SAMPLE (T_0)



350°F/50 HOURS



375°F/50 HOURS

MAGNIFICATION 5000x

FIGURE 23. SCANNING ELECTRON MICROGRAPHS FLSC--Hg LAMP IRRADIATION

CHAPTER 4

CONCLUSIONS

Irradiated solid and solution samples of HNS and HNBiB were found to contain numerous (~15) compounds. The data indicate that photolyzed HNS and HNBiB result in a complex mixture of nitrocompounds similar to (1) compounds observed during thermal degradation,⁶ (2) by-products⁷ isolated from the Shipp HNS synthesis process,^{8,1} (3) compounds isolated from irradiated aqueous solutions of TNT (pink water),⁹ and (4) compounds observed during the thermal degradation of TNT.¹⁰

The photodecomposition products isolated from the sunlight and Hg lamp irradiation of HNS include: 2,4,6-trinitrobenzaldehyde (PiCHO); 4,6-dinitroanthranil (Anil); 1,3,5-trinitrobenzene (TNB); 2,4,6-trinitrobenzoic acid (PiCOOH); and 4,5-dinitroisoanthranil (iso-Anil). The products isolated from the HNBiB photodecomposition include: PiCHO, Anil, TNB, PiCOOH, isoAnil, TNT, trinitrobenzyl alcohol (PiCH₂OH), 2,4,5-trinitrobenzonitrile (PiCN), and HNS. These photoproducts account for about 10 percent of the total photoproducts observed. Approximately 30 percent of the photodecomposition products defied separation and identification due to (1) the trace quantities that were formed, and (2) the instability of the compounds as evidenced by TLC and HPLC. The remaining material (~60 percent) consisted of a dark brown residue and a waxy oil and/or waxy solid substance which contained high molecular weight and/or polar compounds, since most of this material remained at the origin when chromatographed with 100 percent methanol.

Of the two materials (HNS and HNBiB), the data indicate that HNBiB is about five times more photosensitive than HNS. Several of the benzene soluble photoproducts (PiCHO, Anil, PiCH₂OH) were observed to be more photo labile than either HNS or HNBiB. The most stable photoproduct isolated from this study was TNB.

The SMDC's (2-1/2 grains/foot) fabricated from the irradiated (sunlight, Hg lamp) HNS material performed satisfactorily (initiated along the entire length) after additional thermal exposure of 350°F/50 hours. However, all the SMDC lines failed to fully initiate after being exposed to a thermal input of 375°F/50 hours. These failures were randomly distributed along the SMDC's. None of the failures occurred in the booster tips.

In a previous service life evaluation study¹³ of 800 HNS (2-1/2 grains/foot) SMDC lines, the data indicated no explosive propagation failures.

Lines were removed from a variety of military aircraft e.g., F-111, F-14, AH-IG, and the AH-IS. The data show that these lines were not adversely affected by age (up to 10 years), service (up to 7 years), a repeat of their thermal qualification tests, or additional thermal exposure to 375 degrees for 50 hours.

In this current study, neither additional thermal exposure to (375°F/50 hours) nor irradiation (sunlight or Hg lamp) appeared to have any significant effect on the performance parameters of the 20 grains/foot FLSC lines.

The photolytic conditions detailed in this report are generally beyond those which HNS is subject to in normal synthesis and handling operations. Because of the number (8) of lines used for the detonation velocity measurements, no statistical data is available. No determinations were made regarding the influence of age or service life type conditions on the SMDC's or FLSC's containing the photolyzed HNS.

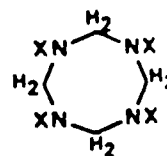
In general, the results of this research indicate that the photodecomposition that the HNS is subjected to in this study does not affect the performance of the HNS-I in the SMDC booster tip; however, it does affect the performance of the HNS-II fabricated into the 2-1/2 grains/foot SMDC line. As the uses for HNS increase (especially small particle HNS-IV), photolysis during synthesis and handling could pose a problem. The smaller the particle size (the larger the surface area), the more critical or damaging the effects of photolysis can become. Therefore, an additional test procedure has been added to the HNS-IV Material Specification¹⁴ to ensure that this material, when used in any man-rated system, will adequately execute its life-critical function.

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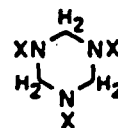
NOMENCLATURE

| | | |
|------------------|---|---|
| Ag | - | silver |
| Anil | - | 4,6-dinitro[2,1]benzisoxazole |
| BT | - | booster tip |
| DMSO | - | dimethyl sulfoxide |
| EDA | - | ethylene diamine |
| FLSC | - | flexible linear shaped charge |
| Hg | - | mercury |
| HNS | - | 2,2'4,4'6,6'-hexanitrostilbene |
| HNS-I | - | explosive material synthesized from trinitrotoluene (TNT) and sodium hypochlorite (NaOCl)--MP 601°F |
| HNS-II | - | material recrystallized from HNS-I in an organic solvent--MP 604°F |
| HNS-IV | - | small particle size HNS-II |
| HMX | - | 1,3,5,7-tetranitro-1,3,5,7-tetra-azacyclooctane |
| HNBiB | - | 2,2'4,4'6,6'-hexanitrobibenzyl--MP 420°F |
| HPLC | - | high-performance liquid chromatography |
| Hz | - | hertz--frequency in cycles/second |
| iso-Anil | - | 4,6-dinitroisoanthranil |
| λ | - | Lambda wavelength |
| LC | - | liquid chromatography |
| man-rated system | - | emergency aircraft crew escape system |
| MP | - | melting point |



NOMENCLATURE (Cont.)

| | | |
|----------------------|---|---|
| nm | - | nanometers |
| NMR | - | nuclear magnetic resonance |
| NODNBA | - | 2-nitroso-4,6-dinitrobenzoic acid |
| OD | - | outside diameter |
| PiCHO | - | 2,4,5-trinitrobenzaldehyde |
| PiCH ₂ OH | - | 2,4,5-trinitrobenzyl alcohol |
| PiCN | - | 2,4,5-trinitrobenzonitrile |
| PiCOOH | - | 2,4,5-trinitrobenzoic acid |
| RDX | - | 1,3,5-trinitro-1,3,5-triazacyclohexane cyclo-1,3,5-trimethylene-2,4,6-trinitramine, cyclonite |
| R _f | - | distance from the origin to the spot/distance from the origin to the solvent front |
| SEM | - | scanning electron microscopy |
| SMDC | - | shielded mild detonating cord |
| TL | - | transfer line |
| TLC | - | thin-layer chromatography |
| TNB | - | 1,3,5-trinitrobenzene |
| TNT | - | 2,4,6-trinitrotoluene |
| v,v | - | volume per volume |
| VTs | - | vacuum thermal stability |



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| Tracon Aerospace Attn: S. Palermo Ballinger Canyon Road San Ramon, CA 94583 | 1 | R10D (H. Adolph) | 1 |
| Space Ordnance Systems Attn: T. Ritche 25977 Sand Canyon Rd. Saugus, CA 91351 | 1 | R11 (D. Carlson) | 1 |
| Rockwell International Attn: L. Corvin (MA2) Los Angeles, CA 90009 | 1 | R11 (C. Gotzmer) | 1 |
| Monsanto Research Corporation Attn: M. R. Fearheller P.O. Box 32 Miamisburg, OH 45342 | 1 | R11 (N. Johnson) | 1 |
| Ford Aerospace Corporation Attn: D. Harrison 3939 Fabian Way Palo Alto, CA 94303 | 1 | R11 (J. Leahy) | 1 |
| | | R11 (V. Ringbloom) | 1 |
| | | R12 (L. Montesi) | 1 |
| | | R12 (P. Spahn) | 1 |
| | | R16 (J. Corney) | 1 |
| | | R16 (J. Deiter) | 1 |
| | | R16 (E. Kayser) | 20 |
| | | R16 (L. Lee) | 1 |
| | | R16 (R. Renner) | 1 |
| | | R34 (M. Norr) | 1 |